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DEVELOPMENT OF TECHNOLOGY FOR THE PRODUCTION OF LIGHTWEIGHT OBSERVATION AND PROTECTIVE CONTAINER (LOOK) MADE OF NANOSTRUCTURED ULTRA-STRENGTH STEELS

OPRACOWANIE TECHNOLOGII PRODUKCJI LEKKIEGO KONTENERA OBSERWACYJNO-OBRONNEGO (LOOK) ZE STALI NANOSTRUKTURALNYCH ULTRAWYTRZYMAŁYCH

The article contains results of research and analyses concerning application of nanostructured bainitic steel in the form of plates for manufacturing of armour components. The presented results of examination of microstructure and properties include a wide range of laboratory experiments and industrial tests, which resulted in the achievement of the assumed functional properties. In the period of 2017-2021, a scientific and industrial consortium consisting of Łukasiewicz – Institute of Ferrous Metallurgy (leader); WITPiS, Tarnów Mechanical Works, Alchemia and Heatmasters Poland carried out a project funded by the POIR 04.01.04 programme aimed to develop the design and to manufacture an observation and protective container with a specified resistance to penetration by armour-piercing projectiles and with a lower mass of steel armouring in relation to that currently produced. The aim of the project was achieved by using armour plates made of nanostructured bainitic steel (nanobainitic), which are characterised by high resistance to

Artykuł zawiera wyniki badań i analiz dotyczące zastosowania stali nanostrukturalnej bainitycznej w postaci blach arkuszowych do wytwarzania elementów opancerzenia. Przedstawione wyniki badań mikrostruktury i właściwości obejmują szeroki zakres eksperymentów laboratoryjnych i badań przemysłowych, w wyniku których osiągnięto założone właściwości użytkowe. W latach 2017-2021 konsorcjum naukowo-przemysłowe w składzie: Łukasiewicz – Instytut Metalurgii Żelaza (lider), Wojskowy Instytut Techniki Panczernej i Samochodowej (WITPiS), Zakłady Mechaniczne Tarnów S.A. (ZMT), Alchemia S.A. i Heatmasters Poland sp. z o.o. zrealizowało projekt finansowany z programu POIR 04.01.04, którego celem było opracowanie konstrukcji i wykonanie kontenera obserwacyjno-obronnego o określonej odporności na przebiecie pociskami przeciwpancernymi oraz o niższej masie opancerzenia stalowego w odniesieniu do obecnie wytwarzanego. Cel projektu osiągnięto przez zastosowanie blach pancernych ze stali nanostrukturalnej

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high-energy impact concentrated in a small area. The technological tests carried out in the project mainly concerned the development of a new container and industrial technology of armour plates production and their application in the armour of this container. Based on the results of investigation of the semi-industrial scale material, the optimum chemical composition for industrial scale melting and casting was determined. An industrial technology for the production of plates of nano-structured bainitic steel was developed, which includes the following processes: smelting and casting, preliminary heat treatment and ingot hot processing, as well as hot rolling, final heat treatment, and surface treatment. A test batch of the material in the form of 1500×2470 mm armoured plates was fabricated under industrial conditions. The final result of the project is a container armoured with bainitic nanostructured steel plates with implementation documentation and a technology for producing armoured plates from this steel under the technical and technological conditions of domestic steel manufacturers.

Keywords: nanostructured bainitic steel, production technology, plates, microstructure, mechanical properties, ballistic protection, observation and protective container

bainitycznej (nanobainitycznej-NBA), które charakteryzuje wysoka odporność na skoncentrowane na małym obszarze wysokoenergetyczne oddziaływanie udarowe. Zrealizowane badania przemysłowe w projekcie dotyczyły w głównej mierze opracowania konstrukcji nowego kontenera oraz przemysłowej technologii wytwarzania blach pancernych i ich zastosowania w opancerzeniu tego kontenera. Na podstawie wyników badań materiału wytworzonego w skali półprzemysłowej ustalono optymalny skład chemiczny dla wytopów przemysłowych. Opracowano przemysłową technologię wytwarzania blach ze stali nanostrukturalnej bainitycznej, która obejmuje następujące procesy: wytapianie i odlewanie, wstępną obróbkę cieplną i przetwarzanie wlewków oraz walcowanie na gorąco blach i finalną obróbkę cieplną oraz obróbkę powierzchniową. Wykonano testową partię materiału w warunkach przemysłowych w postaci pancernych blach arkuszy o wymiarach 1500×2470 mm. Finalnym rezultatem projektu jest kontener opancerzony za pomocą blach ze stali nanostrukturalnej bainitycznej z dokumentacją wdrożeniową oraz technologia wytwarzania blach pancernych z tej stali w warunkach techniczno-technologicznych krajowych wytwórców wyrobów stalowych.

Słowa kluczowe: stal bainityczna nanostrukturalna, technologia wytwarzania, blachy, mikrostruktura, właściwości mechaniczne, ochrona balistyczna, kontener obserwacyjno-obronny

1. INTRODUCTION

Nanostructured bainitic (nanobainitic) steels with a relatively low content of alloying elements – typically in the range of 5–7%¹ – allow obtaining ultra-high strength (above 2 GPa) while maintaining acceptable ductility (elongation and impact strength) for many applications. The first research and theoretical analyses, which indicated the potential application of nano-structured bainitic steels, were carried out by Bhadeshia's team [1–6]. Articles, were also published in the 1990s, covering the subject of nanostructured bainitic steels [7]. The common denominator of the work carried out was the chemical composition of the steel, based on the Fe-C-Si-Mn system and the method of heat treatment, leading to obtaining a carbide-free lathy lower bainite and retained austenite. A number of works concerned the design of new grades of nano-structured bainitic steels in order to improve technological properties and obtain the required set of functional properties. In the field of production technology, much attention was paid to the issue of shortening the time of isothermal transformation, while optimising the temperature of this transformation, e.g. by alloying elements. The temperature and time of isothermal transformation are the key parameters of the technology of manufacturing products made of nanostructured bainitic steels. A separate research problem is the role of retained austenite during the use of nanostructured bainitic steel products. The volume fraction, morphology, distribution, chemical composition and, consequently, thermodynamic and mechanical stability of retained austenite are the main research and technological issues in this field. The first works on nanostructured bainitic steels were carried out for high-carbon high-silicon steel grades [8]. As a result of the applied low temperatures of isothermal annealing (125°C), a bainite microstructure with an average lath width of 50 nm was obtained, and the strength reached

the value of 2.5 GPa. However, attention should be paid to very long isothermal annealing times, amounting to several weeks. In [9] for steel with the chemical composition Fe-0.98% C-1.46% Si-1.89% Mn-1.26% Cr-0.26% Mo, annealing temperatures in the range of 125–325°C and times respectively from more than 60 days to 1–2 days were used. Based on the analysis of the kinetics of isothermal transformation, it was found that at temperatures from 125°C to 200°C, a bainitic transformation takes place. A thermodynamic model was used in conjunction with the results of research on properties and microstructure to optimise the chemical composition of nanostructured bainitic steel [10]. The investigated material consisted of steels with a content of 0.8–0.7% C; 1.8–1.9% Si; 2.2–1.4% Mn; 1.0–1.4% Cr; 0.30–0.24% Mo; 1.31–0.14% Co; 0.85–0.75% Al, which, after austenitisation, were isothermally annealed in the temperature range of 200–300°C. The content of retained austenite was in the range of 2–12%, depending on the temperature and time parameters of the heat treatment. The developed thermodynamic model and the test results confirmed the possibility of obtaining a hardness of 610 HV, strength of 1.5–2.0 GPa with an elongation of 8–15%. The issue of improving the impact strength of medium-carbon nanostructured bainitic steel with the following chemical composition: Fe-0.55% C-1.95% Mn-1.82% Si-1.29% Cr-0.72% Mo is described in [11]. The authors proposed a two-stage isothermal annealing, with the first stage below the M_s temperature, in order to partially transform austenite into martensite. The second stage of isothermal annealing was carried out at standard temperature, higher than M_s . The results of Charpy-V impact strength measurement showed a beneficial effect of the presence of martensite laths, which was tempered during the second annealing stage. Impact strength increased, depending on the test temperature, by approx. 50–70% in relation to the variants of the standard treatment, consisting in annealing at a constant temperature of isothermal transformation, higher than M_s . The authors of [12] conducted research on nanostructured bainitic steels with a wide chemical composition: C from

¹ The content of elements was given in weight %, unless specified otherwise

0.49 to 1.00%; Mn from 1.82 to 2.39%; Si from 1.57 to 2.0%; Cr from 0.01 to 1.37%; Co from 0.0 to 1.70%; Mo from 0.27 to 0.80% with microalloying elements V, Ti and Al. The results of measurement of mechanical properties, microstructure studies and analyses of the manufacturing process of nanostructured bainitic steels, under the conditions of mass production, indicated the following optimal chemical composition: 0.56%C-2.0%Mn-1.76%Si-1.29%Cr-0.76%Mo as well as an isothermal transformation temperature of 225°C and time of 60–72 hours. Tensile strength (*TS*) of 1.9 GPa; yield strength (*YS*) of 1.3–1.4 GPa; total elongation (*TE*) of 15% and hardness of 600 HV were achieved. The kinetics of isothermal transformation below the M_s temperature are the subject of [13]. The authors showed that after cooling below M_s , lamellae (or lath packets) of athermal martensite are formed, the fraction of which increases with lowering temperature. The isothermal resistance below the M_s temperature causes the transition of some austenite to bainite and possibly to isothermal martensite. For the tested bainitic nanostructured steels with a carbon content of 0.55% and 0.61%, a clear effect of shortening the time to start the isothermal transformation at a temperature below M_s was obtained, compared to the process which takes place at a temperature higher than M_s . The results of research on nanostructured bainitic steels and methods of producing armour plates from these steels are presented in [14]. Steels with two chemical compositions were tested: Fe-0.80%C-2.01%Mn-1.59%Si-0.24%Mo-1.0%Cr-1.51%Co and Fe-0.79%C-1.98%Mn-1.56%Si-0.24%Mo-1.01%Cr-1.51%Co-1.01%Al. The heat treatment parameters were as follows: austenitising temperature 900°C, cooling in salt and direct isothermal annealing at 250°C and 275°C for 6 and 12 hours. After annealing at 250°C for 12 hours, the material obtained strength of 2.0 GPa and elongation of 15%. The author of the work showed that the austenite deformation of 25% and 35% before isothermal transformation slightly slows down the formation of bainite. The specific features of nanostructured bainitic steel, important for the industrial production process, are presented in [15] on the example of the chemical composition of Fe-0.55%C-1.95%Mn-1.82%Si-1.29%Cr-0.72%Mo. The authors indicated the features of semi-finished products at individual stages of production: from casting, through plastic working, to final heat treatment – isothermal annealing. They discussed, among others, the tendency of nanostructured bainitic steels to interdendritic segregation during casting and its consequence, microstructure banding in hot rolled products. At Łukasiewicz – Institute of Ferrous Metallurgy, the research on this type of steel began in 2008 in a research and development project financed from structural funds [16]. As a result of the project, the chemical composition and method of producing a grade of nanobainitic steel under the name NANOS-BA® [17] were patented. The next stage of the research was the implementation of the project including the phase of preparation for implementation, for the commercial use of NANOS-BA® steel plates in armour components [18]. The test results quoted on the basis of the literature data and the analysed aspects concerning the production of nanobainitic steel products indicate that there is a potential for the use of this grade in conditions of high-energy impact loads.

In the aspect of the presented test results, in particular the achieved values of strength and plastic properties as well as impact toughness, the project – the results of which are included in this article – included experiments on a laboratory and industrial scale aimed at selecting the

parameters for the manufacture of nanobainitic steel products for use in the ballistic shields of the observation and defence container. The armoured observation and protective container is intended for securing and protecting soldiers or officers performing operational tasks during the peacetime as well as during military or stabilisation operations (e.g. at security checkpoints, bases or other strategic facilities, airports, borders, etc.). A container's armour is an important parameter that often determines the possibility of its use. The use of nanostructured steel plates allowed for the improvement of two operational parameters of the container: an increase in the degree of ballistic protection, thus increasing the safety of the crew, and reducing the total weight of the object. Nanobainitic steel is characterised by a high resistance to high-energy impact. The result of this type of interaction may be the perforation of the plate, its cracking or local weakening, contributing to the loss or reduction of the protective capacity. The plates used in the conditions of fire should be characterised by resistance to penetration of certain types of shells, in particular in the conditions of multi-hit fire. The innovative grade of bainitic nanostructured steel is characterised by a high ability to absorb and dissipate impact energy, and the changes in material properties at the point of firing occur in a very small volume, therefore it meets the requirements of the multi-hit firing test.

The scope of work included the design and construction of an innovative container, development of industrial technologies for the production and processing of armour plates made of bainitic nanostructured steel and testing the properties of these plates, including under high-energy dynamic loads. As part of the technological issues related to armour plates, an industrial technology for the production of plates made of nano-structured bainitic steel was developed, which includes the following processes: smelting and casting, preliminary heat treatment and ingot processing, as well as hot rolling, final heat treatment, and surface treatment. During the implementation of the various stages of the project, especially regarding the technology of armour plate production, a number of experiments and tests were carried out, the key of which are described in this article.

2. THE INVESTIGATED MATERIAL, SCOPE AND METHODOLOGY

2.1. LABORATORY MATERIAL

The investigated material consisted of flat bars made of 150×60×800 mm flat nanostructured bainitic steel laboratory ingots in four variants of chemical composition. The rolling of flat bars with a nominal thickness of 6, 7, 8, 10 and 12 mm was carried out using the LPS/B-IMŻ line with a two-high reversing rolling mill with rolls with a diameter of 550 mm. The flat bars were used to produce samples for dilatometric tests, for microstructure and mechanical properties tests, and for firing tests. The material characteristics of nanostructured steels were determined in terms of: CCT diagrams, mechanical properties, hardness, microstructure, including the content of retained austenite. When developing chemical compositions, particular attention was paid to the following issues related to the functional properties and technology of production of nanostructured steel plates for armour: obtaining high strength (above 2 GPa) while maintaining good plasticity (elongation

Table 1. Chemical composition of nanostructured bainitic steel laboratory heats, [weight %]**Tabela 1. Skład chemiczny wytopów laboratoryjnych stali nanostrukturalnej bainitycznej, [% masowe]**

Series heat No.	Requirements type of analysis	C	Mn	Si	P	S	Cr	Mo
1	min	0.60	2.00	1.60	0.000	0.000	1.30	0.50
	max	0.64	2.20	1.75	0.010	0.010	1.40	0.60
	recommended	0.62	2.10	1.65			1.35	0.55
617	melting	0.63	2.10	1.68	0.006	0.011	1.38	0.56
	control	0.65	2.05	1.75	0.005	0.013	1.34	0.57
2	min	0.56	1.65	1.50	0.000	0.000	1.25	0.40
	max	0.60	1.80	1.65	0.010	0.010	1.40	0.50
	recommended	0.58	1.70	1.55			1.35	0.45
640	melting	0.58	1.69	1.57	0.009	0.008	1.35	0.44
	control	0.60	1.67	1.61	0.008	0.011	1.33	0.45
3	min	0.50	2.00	1.65	0.000	0.000	1.45	0.70
	max	0.54	2.20	1.75	0.010	0.010	1.55	0.80
	recommended	0.52	2.10	1.70			1.50	0.75
641	melting	0.53	2.10	1.70	0.009	0.010	1.49	0.75
	control	0.57	2.18	1.71	0.008	0.011	1.48	0.77
4	min	0.48	2.00	1.70	0.000	0.000	1.45	0.70
	max	0.52	2.20	1.80	0.010	0.010	1.55	0.80
	recommended	0.50	2.10	1.80			1.50	0.75
648	melting	0.51	2.10	1.75	0.006	0.008	1.48	0.73
	control	0.50	2.12	1.84	0.004	0.009	1.51	0.81

in static tensile test of at least 12%) and fracture toughness (impact strength KV at room temperature of at least 12 J for 10×10×55 mm sample); the possibility of cooling plates with a thickness in the range of 6–8 mm from austenitising temperature to isothermal transformation temperature, freely in the air or in an accelerated manner with the use of fans, obtaining the lowest possible alloying content (Mn, Si, Cr and Mo) and striving to shorten the time of isothermal heating as much as possible. It is not always possible to meet all the mentioned conditions simultaneously. The key criteria for selecting the material were the results of the mechanical properties tests and the firing tests. Table 1 presents the chemical composition of nanobainitic steel laboratory heats. Laboratory ingots made using the method of vacuum melting and casting met the requirements

for the content of basic elements decisive for obtaining the final functional properties. The ingots also met the requirements for surface quality, shrinkage cavity size and extent, total weight and geometry (dimensions and shape) and were intended for further processing. Homogenisation heat treatment was not used.

2.2. INDUSTRIAL MATERIAL

2.2.1. Nanobainitic steel smelting and casting

Table 2 presents guidelines for the production of industrial heats regarding the chemical composition, and Table 3 presents the results of the chemical composition analysis of the test heats. In the industrial process involving electric

Table 2. Guidelines for the chemical composition of industrial heats of nano-structured bainitic steel, [weight %]**Tabela 2. Wytyczne w zakresie składu chemicznego wytopów przemysłowych stali nanostrukturalnej bainitycznej, [% masowe]**

Steel grade identification		C	Mn	Si	P	S	Cr	Cu	Mo	Al _{met}	N ppm	O ppm
P1	min	0.58	1.65	1.60	-	-	1.35	-	0.55	0.015	max	max
	max	0.62	1.75	1.70	0.010	0.010	1.45	0.15	0.65	0.025	60	25
	Aim	0.60	1.70	1.65	*	*	1.40	*	0.60	0.020	*	*
P2	min	0.54	1.80	1.70	-	-	1.45	-	0.70	0.015	max	max
	max	0.58	1.90	1.80	0.010	0.010	1.55	0.15	0.80	0.025	60	25
	Aim	0.56	1.85	1.75	*	*	1.50	*	0.75	0.020	*	*

*) the lowest possible content in the applied technological process

Table 3. Chemical composition of industrial test heats P1-882917 and P2-883040. The heat analysis was carried out at Alchemia and Łukasiewicz – IMŻ, [weight %]**Tabela 3. Skład chemiczny przemysłowych wytopów badawczych P1-882917 i P2-883040. Analizę wytopową wykonano w Alchemii oraz w Łukasiewicz – IMŻ, [% masowe]**

Heat No. identification	Analysed by	C	Mn	Si	P	S	Cr	Mo	Al	Cu	N ppm	O ppm
882917 P1	ALCH	0.60	1.69	1.65	0.010	0.004	1.41	0.59	0.023	0.07	32	9
	Ł-IMŻ 1	0.59	1.70	1.60	0.012	0.004	1.40	0.59	0.022	0.07	27	17
	Ł-IMŻ 2	0.59	1.69	1.60	0.012	0.004	1.40	0.59	0.022	0.07	24	7
883040 P2	ALCH	0.56	1.85	1.75	0.013	0.003	1.49	0.74	0.026	0.09	23	16
	Ł-IMŻ 1	0.55	1.81	1.74	0.017	0.004	1.46	0.75	0.025	0.093	26	10
	Ł-IMŻ 2	0.56	1.82	1.74	0.017	0.004	1.47	0.75	0.025	0.093	28	11

arc furnace (EAF) smelting, secondary vacuum furnace treatment (VAD) and uphill casting, the main elements (C, Mn, Si, Cr, Mo) were at the correct level, and only the phosphorus content was slightly exceeded. A material in the form of ingots from two nanostructured bainitic steel heats were produced. While cooling after casting, the ingots, after reaching the temperature of approx. 700°C, were placed in a heating furnace and subjected to softening and annealing at 720°C. A low hardness of approx. 260 HB was achieved on the surface. After soft and stress-relief annealing, the material was intended for forging into flat slabs.

2.2.2. Nanobainitic steel ingot forging

The ingots were forged into 120×800×1350 mm slabs which constituted the feedstock for the rolling mills. The ingots were heated with the furnace to 1200°C, and then held at this temperature for 30 hours in order to reduce the degree of segregation. During forging, the temperature range of 1200–900°C was used with an acceptable temperature drop in the area of the corners and the surface layer to approx. 850°C. The slabs were transported to the furnace immediately after forging and cooled down with the furnace to ambient temperature. Then, the slabs were subject to the process of cold straightening. No surface defects were found that could significantly affect the further process of plastic working using hot rolling. The slabs met the requirements for geometry and surface quality for further processing. In the field of the preliminary heat treatment process and hot forging of bainitic nanostructured steel at ALCHEMIA S.A. the highest level of technological readiness was achieved.

2.2.3. Hot rolling of nanobainitic steel plates

Industrial trials of hot rolling of nanobainitic plates was carried out, as a result of which 1500×2470 mm plates with a thickness in the range of 6–9 mm were produced. The

**Fig. 1. Photograph of a strip during hot rolling****Rys. 1. Fotografia pasma w trakcie walcowania na gorąco**

plates, immediately after rolling and cooling in the air to the temperature on the surface of approx. 200–300°C, were stacked and cooled down to ambient temperature. Due to the high hardenability of the steel resulting in a hardness of 60 HRC (approx. 735 HV), the material in this state could not be cut with guillotine shears. A sample was taken from 6 mm thick plates from heat P1 after hot rolling for preliminary material tests. In order to reduce hardness, the plates were subjected to soft annealing at 680°C. After softening, the hardness was 350 HB. Fig. 1 presents photographs of the plates during rolling.

2.2.4. Final heat treatment of plates in industrial conditions

Heat treatment of nanostructured bainitic steels is a key production stage. The process consists of several immediately consecutive stages: heating, austenitising, controlled cooling and isothermal annealing, as well as cooling to ambient temperature. Preliminary cycles of heat treatment were performed using the following parameters of the individual stages: austenitising at 950°C, controlled cooling with air blowing to cool the plates at a rate of min. 1°C/s and isothermal annealing in the temperature range of 210–225°C. As a result of examining the microstructure and mechanical properties as well as firing tests carried out on steel plates, the following parameters of the final heat treatment were determined: austenitising temperature of 950°C and time of 30 minutes, accelerated cooling with the use of two mobile fans and isothermal annealing immediately after cooling at two temperatures at 225°C for 12 hours and then at 210°C for a total of both temperatures – including the time needed to reach 210°C – for 96 hours (Fig. 2). The time of cooling from higher to lower temperature was approx. 1 hour. Plates with a thickness of 7–9 mm were produced in such manner. “Witness” tensile samples were used in each research bundle (cycle). A research batch of plates was produced and components of the container armour were prepared in the form of plate formats with the assumed functional properties, dimensions and surface quality.

2.3. TESTING METHODS

The microstructure of nanobainitic steels was examined using a light microscope (LM), scanning electron microscope (SEM) and transmission electron microscope (TEM). The EBSD method in SEM was used to investigate the morphology, arrangement, size distribution and volume fraction of blocky retained austenite. The content of retained austenite was also measured with an X-ray diffractometer

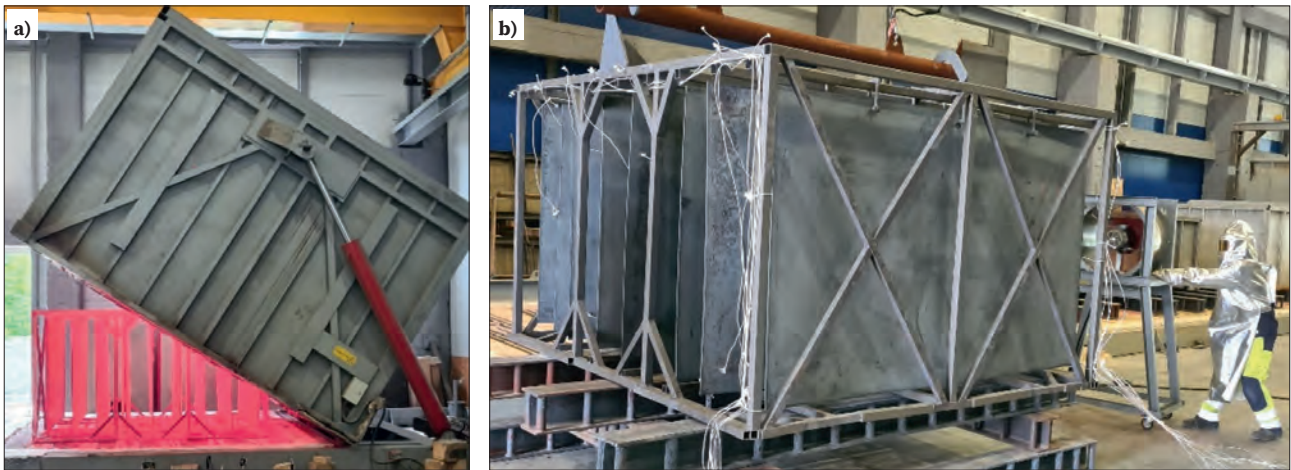


Fig. 2. Photograph of plates during austenitising (a) and controlled cooling (b)

Rys. 2. Fotografia blach podczas austenitzowania (a) i regulowanego chłodzenia (b)

using filtered cobalt radiation in the configuration with a Pixcel detector. The morphology of retained austenite in the form of laths was studied using TEM. A static tensile test was used to determine mechanical properties (TS , YS , TE). Charpy-V impact strength was measured on $7.5 \times 10 \times 55$ mm samples at -60 to $+40^\circ\text{C}$. The nominal height of the sample below the notch was 8 mm. The hardness measurement was carried out using the HRC, HV and HB methods. Additionally, uniaxial compression tests of cylindrical samples with strain rates up to 100 s^{-1} were carried out using a Gleeble 3800 GTC simulator. The firing tests for the plates were carried out at WITPiS with the use of ballistic barrels. The barrel's quadrant elevation was 0° and 30° . The shell impact velocity was measured using a PVM-2008/21 chronograph. A witness plate, i.e. a 0.5 mm thick aluminium plate, was placed behind the sample at a distance of 150 mm. The firing was carried out at ambient temperature, without the samples' temperature being controlled, and the number of shots was determined during the tests depending on the subsequent test results.

3. RESULTS OF INVESTIGATION FOR LABORATORY MATERIAL

3.1. RESULTS OF MICROSTRUCTURE EXAMINATION AND MEASUREMENT OF MECHANICAL PROPERTIES

In the first stage of testing the laboratory material, dilatometric measurement and heat treatment tests were carried out in order to determine the possibility of obtaining the desired mechanical properties. The dilatometric tests included the determination of characteristic temperatures, the development of a part of the CCT phase diagrams (an example for the laboratory melt is presented in Fig. 3) and the final heat treatment including austenitisation, controlled cooling and isothermal annealing in the temperature range of 180 – 240°C for up to 120 hours. Under the conditions of laboratory heat treatment of tensile samples, the required degree of similarity to the industrial research planned at a later stage regarding the final heat treat-

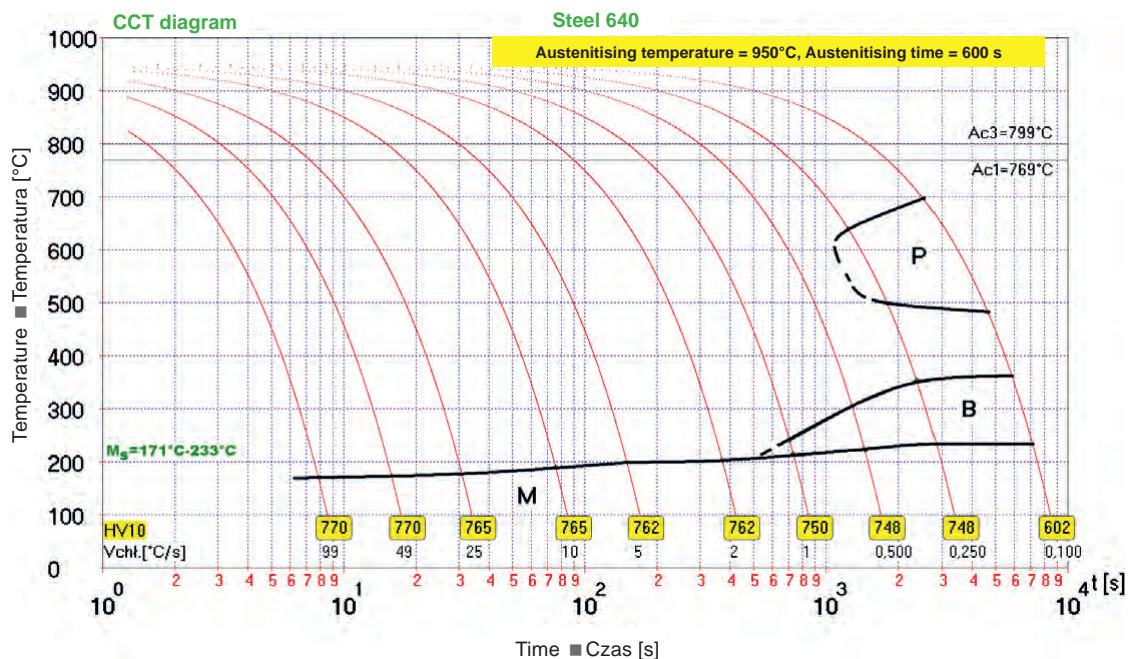


Fig. 3. CCT diagram for nanostructured bainitic steel – heat 640

Rys. 3. Diagram CTPc stali nanostrukturalnej bainitycznej – wytop 640

ment of plates was maintained. M_s temperature changes for a wide range of cooling rate are marked in Fig. 3. The kinetic curves indicate that, for the time range used, the transformation takes place to a large extent at 210–240°C. After isothermal annealing, the samples were subjected to microstructure tests and hardness measurement. As a result of isothermal annealing at temperatures of 210–240°C, a typical microstructure of carbide-free nanolath bainite and retained austenite were obtained, similar to the results of [10–14]. Based on the results of dilatometric tests, experiments of heat treatment of tensile and impact samples were carried out. The samples were heated to 950°C and annealed for 30 minutes and cooled in air to the temperature of isothermal transformation. Then, direct isothermal annealing was applied in the temperature range of 200–240°C for 72 to 137 hours.

Hardness measurement and microstructure results of investigation were performed on the non-deformed parts of the samples. Fig. 4 presents typical images of the tested samples' microstructure. Depending on the temperature of isothermal transformation, the steel microstructure consists of lath packets of carbide-free bainite and retained austenite, and in the case of transformation temperature below M_s , additional lathy martensite is formed, which is tempered. The value of the isothermal annealing temperature affects the width of bainite laths and the content and form of retained austenite, in such a way that the higher the transformation temperature, the greater the width of the laths and the greater the content of retained austenite. The principle mentioned above, due to the heterogeneity of the material, typical for the production conditions related to the casting method, and slight differences in the applied temperatures of isothermal annealing, is not always met. The results of measurement of the austenite content for laboratory heats and selected variants of heat treatment are presented in Table 4. The preliminary correlation analysis of the austenite content and the mechanical and functional properties of the material indicates that the optimal volume fraction of retained austenite should not exceed

20%, and most preferably be within the range of 13–18%. The results of the measurement of mechanical properties in a static tensile test are presented in Table 5. Taking into account the values of mechanical properties, the shape of the tensile curves in terms of strengthening and the value of the TS/YS ratio, variants were selected for the production of plates for firing tests. Mechanical properties assumed in the project: TS min. 2000 MPa, TE min. 12% and impact strength KV min. 12 J at ambient temperature were met for the developed chemical compositions of steel and most of the applied heat treatment variants.

Table 4. Results of retained austenite content measurement. Laboratory heat 640/XRD method

Tabela 4. Wyniki pomiarów zawartości austenitu reszkowego. Wytóp laboratoryjny 640/Metoda XRD

Sample identification (heat treatment variant – temp., [°C]/time, [h])	Retained austenite volume fraction, [%]
210/116	13.0
210/120	15.2
	13.9
215/96	10.4
220/72	21.0
	17.5
220/94	13.7
	13.8
220/96	12.1
	15.3
225/70	17.8
235/72	16.9
235/100	13.7

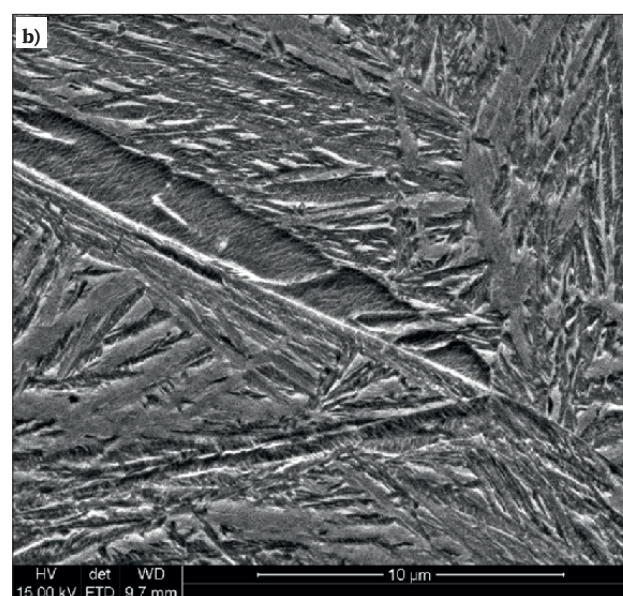
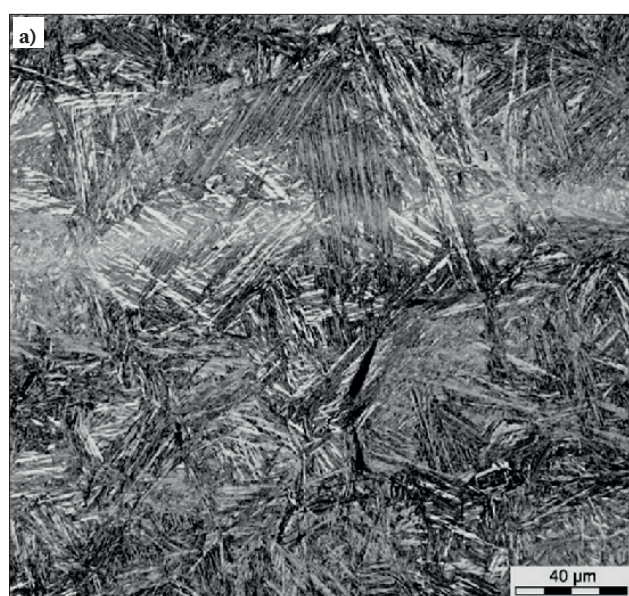


Fig. 4. Microstructure of NBA steel-heat 640 after isothermal treatment: a) light microscope, b) scanning electron microscope (220/96)

Rys. 4. Mikrostruktura stali NBA-wytóp 640 po obróbce izotermicznej: a) mikroskop świetlny, b) skaningowy mikroskop elektronowy (220/96)

Table 5. Results of measurement of mechanical properties in static tensile test. Laboratory heats**Tabela 5. Wyniki pomiarów właściwości mechanicznych w statycznej próbie rozciągania. Wytopy laboratoryjne**

Heat No. – identification of isothermal annealing variant (temp., [°C]/time, [h])	YS [MPa]	TS [MPa]	TE [%]	UE* [%]	TS/YS
617-210/137	1366	2165	13.1	11.2	1.58
	1279	2064	8.7	4.4	1.61
617-230/96	1354	2022	12.4	10.2	1.49
	1330	2008	14.4	10.4	1.51
640-210/116	1470	2072	11.5	8.7	1.41
	1467	2097	12.5	9.0	1.43
640-210/120	1497	2109	8.0	7.7	1.41
	1451	2107	12.2	8.3	1.45
640-215/96	1455	2068	11.7	8.3	1.42
	1457	2091	12.5	8.6	1.44
640-220/72	1436	2132	11.6	8.2	1.48
640-220/94	1516	2095	10.4	9.1	1.38
	1533	2112	10.7	8.6	1.38
640-220/96	1485	2081	13.1	8.0	1.40
	1535	2106	12.2	8.1	1.37
641-210/116	1154	2087	14.0	11.4	1.81
	1242	2059	14.0	10.8	1.66
641-210/120	1164	2102	14.0	11.9	1.81
	1168	2096	12.7	11.5	1.79
648-215/96	1244	2000	14.4	11.1	1.61
	1265	1996	10.7	10.2	1.58

*UE – uniform elongation

3.2. FIRING TEST RESULTS

Firing tests of material from laboratory heats were carried out with the use of 7.62×51 mm API BZ ammunition and, additionally, 5.56×45 mm M193 ammunition. Two plate sections (flat bars) from laboratory heats 617, 640, 641 and 648 were prepared for the firing tests. Firing tests were

carried out at an angle of 0° for the actual thickness in the range of 7.0–8.5 mm and at an angle of 30° for plate thickness in the range of 5.3–6.3 mm. Based on the observation immediately after the firing, no cracks were found on the plate as a result of multi-hit firing. Typical test results and photographs of plates after the firing are presented in Table 6 and Fig. 5, respectively.

Table 6. Firing test results for laboratory heat plates**Tabela 6. Przykładowe wyniki testów ostrzałem blach z wytopów laboratoryjnych**

No. (test No. – Fig. 5)	Heat / HT parameters (temp., [°C], time, [h])	Ammunition type, firing angle, plate thickness	Impact velocity, [m/s]	Test result NP – no perforation, P – perforation
1	640 (215/96)	7.62×39 mm API BZ (0°) #7.5 mm	718.2	NP
2			713.1	P
3			714.1	NP
4	640 (215/96)	5.56×45 mm M193 (0°) #7.5 mm	943.6	NP
5			940.2	NP
6			949.2	NP
1	640 (220/71)	5.56×45 mm M193 (30°) #6.3 mm	934.8	NP
2			946.2	NP
3			939.2	NP
4			931.7	NP
5	640 (220/71)	7.62×39 mm API BZ (30°) #6.3 mm	707.7	NP
6			706.1	NP
7			710.8	NP
8			703.4	NP

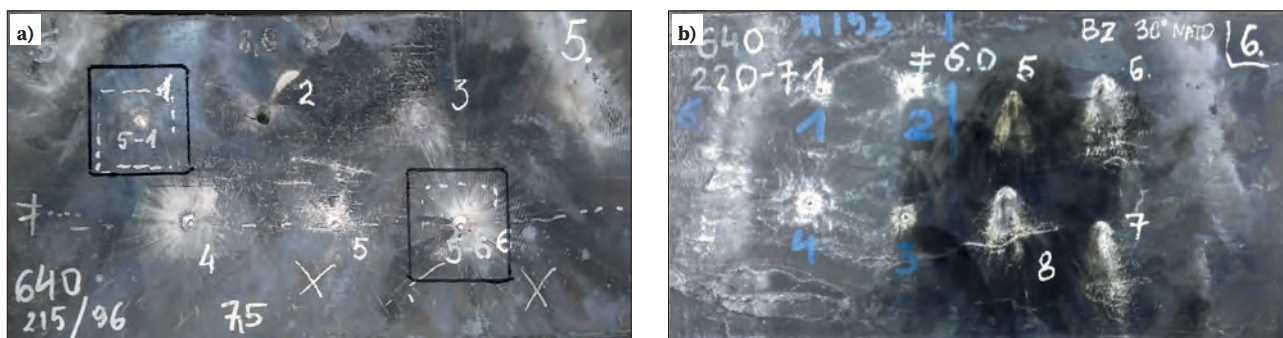


Fig. 5. Photographs of nanostructured bainitic steel plates after firing. Laboratory heat 640

Rys. 5. Fotografie blach ze stali bainitycznych nanostrukturalnych po ostrzale. Wytop laboratoryjny 640

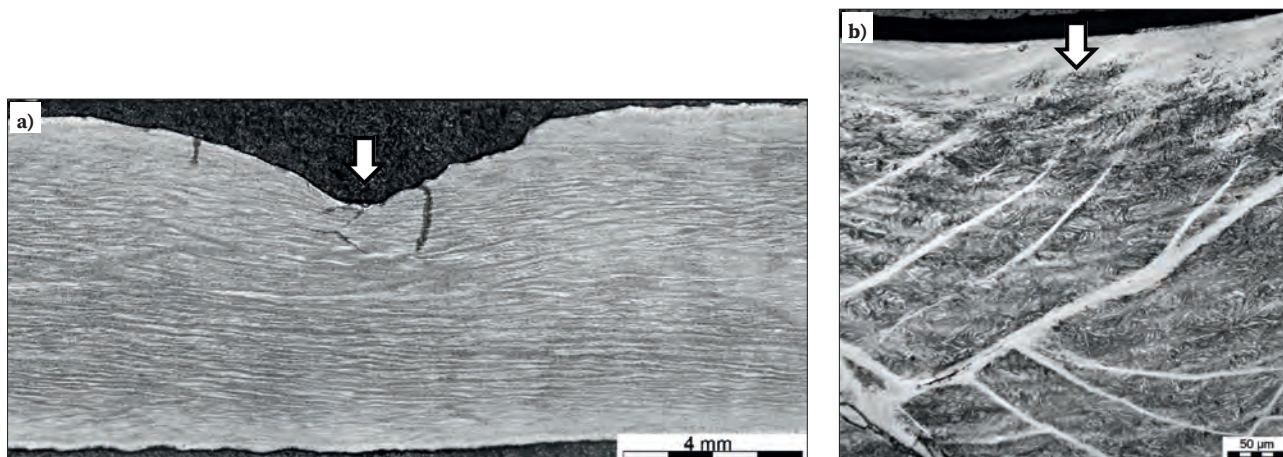


Fig. 6. Sample results of : a) macro- and b) microstructure tests in the area of the projectile core-plate interaction, heat 640, marked direction of the projectile's trajectory

Rys. 6. Przykładowe wyniki badań: a) makro- i b) mikrostruktury w obszarze oddziaływania rdzenia pocisku z blachą, wytop 640, zaznaczono kierunek lotu pocisku

For selected variants of heat treatment and test parameters, microstructure tests were performed at projectile impact sites. The purpose of the tests included the assessment of the degree of degradation and depth of penetration. Based on the test results, a comparative analysis of the materials was carried out in terms of resistance to perforation, including in terms of the occurrence of shear bands, macro- and microcracks and the bullet core penetration depth or the degree of knocking out of the “plug” (also the tendency to the formation of secondary shrapnel). Two typical penetration mechanisms were observed: adiabatic shear for the M193 projectile and erosion for the incendiary AP projectile (Fig. 6). The assessment of the degree of degradation of the firing locations was used in the final stage of selecting the chemical composition and parameters of material production on an industrial scale. The results of the research indicated, among others, the influence of the segregation of elements formed during solidification – and, consequently, the banding of the microstructure – on the resistance to perforation of the plates.

4. TEST RESULTS OF MATERIAL PRODUCED IN INDUSTRIAL CONDITIONS

4.1. PRELIMINARY RESULTS OF THE STUDY OF MICROSTRUCTURE AND MECHANICAL PROPERTIES

The preliminary tests were carried out on a 6 mm thick plate section from heat P1 after hot rolling. The scope of

the tests included microstructure characteristics and hardness measurement. Subsequently, heat treatment of softening annealing and austenitising, as well as a final treatment including austenitising and direct isothermal annealing were performed. The microstructure of the plates after rolling and slow cooling in a stack was composed of martensite, bainite and pearlite (Fig. 7a). There was a microstructure banding typical of hot-rolled plates, with bands distributed evenly on the thickness (Fig. 7b). The results of the plate's surface quality examination showed that the thickness of the oxidised layer was approx. 20 μm. Despite cooling, the stacked material reached the hardness of approx. 60 HRC. Heat treatment was carried out to soften the steel. A temperature of 690°C and cooling with the furnace were applied. The microstructure of the steel after this heat treatment, causing recrystallisation of the matrix and the precipitation and growth of carbides, was made of ferrite and partially coagulated carbides with a diameter below approx. 200 nm (Fig. 7c). As a result of the softening, the hardness of the material was reduced to 33 HRC (318 HV). In the next stage of the study, the austenitising process was analysed, as a result of which a homogeneous austenite with the smallest grain size should be obtained (without carbides that should dissolve). For this purpose, heat treatment was carried out with two temperatures of 930 and 955°C and a holding time of 30 minutes. After heat treatment, microstructure results of investigation were performed in order to verify the correctness of the parameters used, based on the presence of primary precipitates and grain size. After austenitising at 930°C, the presence of single Cr and Mo carbides was observed. After austenitising at

955°C, carbides smaller than 200 nm were sporadic. Based on the research, the austenitising temperature in the final heat treatment process was determined to be 950°C and the holding time at this temperature was 30 minutes. In the last stage of the preliminary tests, the final heat treatment was carried out, consisting in isothermal annealing immediately after austenitisation and controlled cooling. Based on the tests carried out on the material produced in laboratory conditions, an isothermal annealing temperature of 215°C and a time of 96 hours were used. The transverse tensile samples (perpendicular to the rolling direction of the plates), which were the material for the study of microstructure and mechanical properties, were processed. A typical tensile curve is presented in Fig. 7d. The assumed properties were obtained: tensile strength higher than 2000 MPa and elongation above 12%, with a yield strength of approx. 1500 MPa. During quasi-static tensile tests, the samples exhibited a distinct narrowing and the ductile nature of the fracture surface. In the areas of the holders of strength samples, microstructure studies were performed using a scanning electron microscope, which were nanobainite packets without the presence of carbides.

4.2. RESULTS OF DILATOMETRIC TESTS

Figs. 8 and 9 present CCT phase transformation diagrams for industrial test heats. The following temperatures characteristic for heating were determined based on the analysis of dilation curves. P1: $A_{c1} = 747^\circ\text{C}$; $A_{c3} = 847^\circ\text{C}$; P2: $A_{c1} = 750^\circ\text{C}$; $A_{c3} = 848^\circ\text{C}$. The austenitising temperature used before isothermal annealing for samples from both test heats was 950°C. The experiments and studies of the austenitising process carried out so far have confirmed that annealing at the temperature of 950°C for 30–60 minutes guarantees obtaining a homogeneous austenite solution without the presence of carbides and does not lead to excessive growth of austenite grains. M_s temperatures for industrial heats were determined after austenitising at 950°C for 600 s and with cooling with a rate of 1–100°C/s. The cooling rate applied in practice is within the following range: 1–5°C/s. For this range of cooling rate, the M_s temperature ranges from 201 to 220°C for heat P1 and from 197 to 205°C for heat P2.

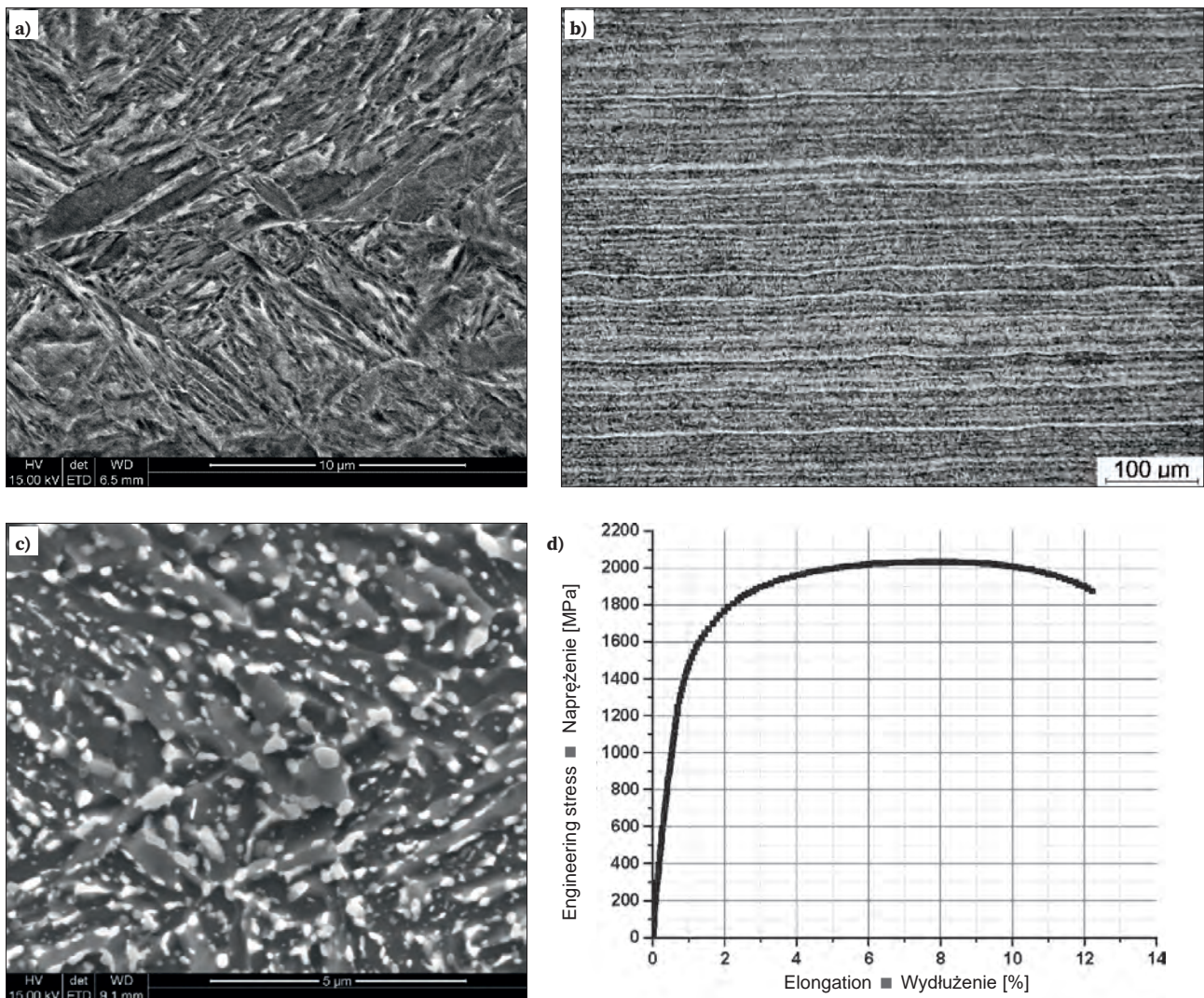


Fig. 7. Microstructure of nanostructured bainitic steel after rolling (a, b) after softening annealing at 690°C for 6 hours (c) and tensile curve of nanobainite steel for the 215°C/96 hours variant. Heat P1, transverse samples (perpendicular to the rolling direction) (d)

Rys. 7. Mikrostruktura stali bainitycznej nanostrukturalnej po walcowaniu (a, b) po wyżarzaniu zmiękczającym w temperaturze 690°C przez 6 godzin (c) oraz krzywa rozciągania stali nanobainitycznej dla wariantu 215°C/96 godzin. Wytop P1, próbki poprzeczne (prostopadłe do kierunku walcowania) (d)

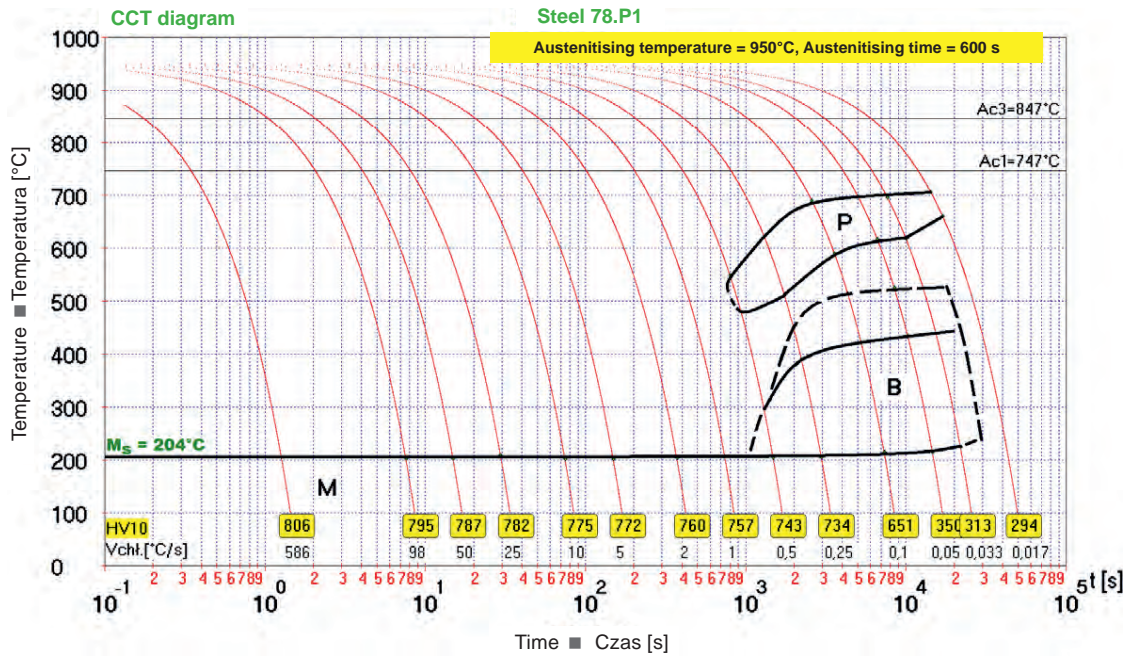


Fig. 8. CCT diagram for nanobainitic steel. Heat P1
 Rys. 8. Diagram CTPc stali nanobainitycznej. Wytóp P1

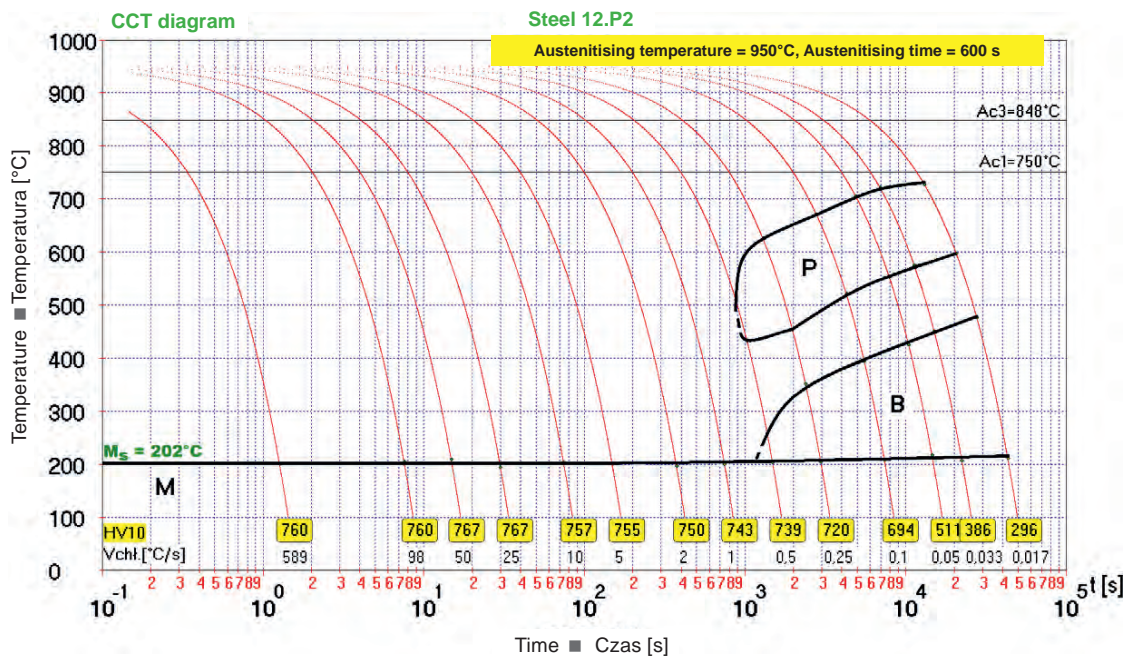


Fig. 9. CCT diagram for nanobainitic steel. Heat P2
 Rys. 9. Diagram CTPc stali nanobainitycznej. Wytóp P2

4.3. TEST RESULTS AFTER FINAL HEAT TREATMENT IN LABORATORY FURNACES

Before the guidelines for the technology of heat treatment of plates were developed, a wide range of tests and analyses of industrial material was carried out, which was subjected to heat treatment at Łukasiewicz – IMŻ. Characteristics in terms of microstructure and mechanical properties were developed for this material, and its protective effectiveness was assessed in firing tests. The test results were the basis for indicating the conditions for industrial heat treatment, which is a key stage of technology, shaping the functional properties.

4.3.1. Results of microstructure examination

Fig. 10 presents microstructure images of nanobainitic steel after final heat treatment. The range of magnifications used in the light microscope enables the observation of the following features characterising the material: banding, surface oxidation and decarburisation, non-metallic inclusions larger than approx. 1 μm, microstructure components up to a specific level of detail. In the case of nanobainitic steel plates, there is microstructural banding, and the distance between adjacent bands is from 10 to 20 μm. The microstructure of the material after the final heat treatment consists of packets of nanobainite laths. At high-

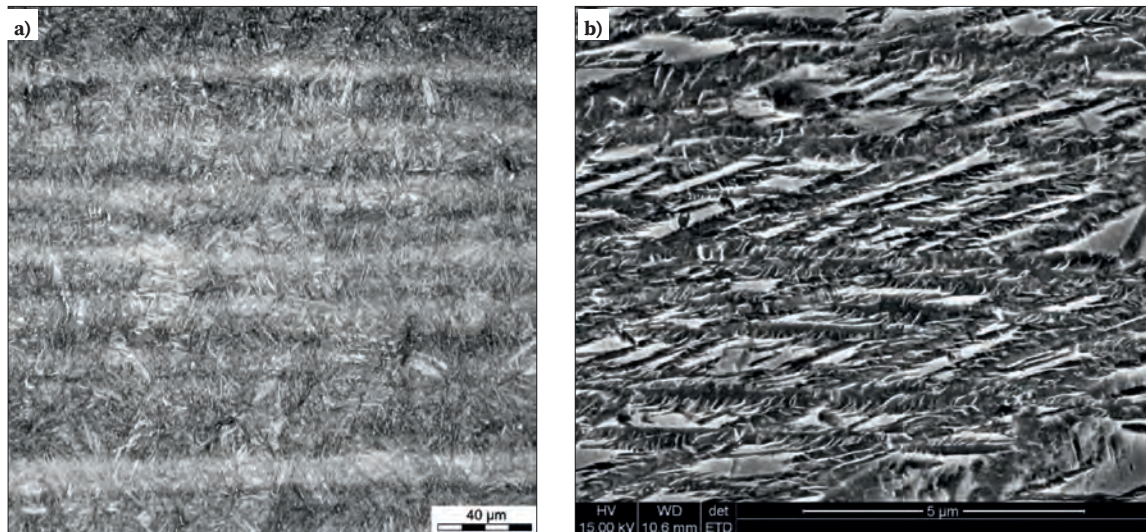


Fig. 10. Microstructure on longitudinal microsection of P2 heat plate – variant 215/96. Light microscope (a), scanning electron microscope (b)
Rys. 10. Mikrostruktura na przekroju wzdłużnym blachy z wytopu P2-wariant 215/96. Mikroskop świetlny (a), skaningowy mikroskop elektryczny (b)

er magnifications, in particular in the areas of segregation with a higher content of elements than the average composition (in light bands), individual grains of blocky retained austenite can be observed (Fig. 10a). A detailed analysis of the retained austenite morphology can be assessed using a scanning microscope (blocky austenite – A_B) and a transmission electron microscope (lathy austenite – A_L). Fig. 10b presents the results of investigation of the microstructure of nanobainitic steel from the P2 heat – variant 215/96 – using SEM. The microstructure consists of laths of nanobainite. Slight differences in nanobainite morphology were observed in the segregation (banded) areas. A typical microstructure consisting of nanobainite laths was present in areas depleted in alloying elements (Mo, Cr, Mn, Si). In areas enriched with the above-mentioned alloying elements, a higher content of components in the form of blocks, including retained austenite, was observed. A clear identification of blocky retained austenite using observation with the applied magnifications and the method of revealing (etching) the microstructure is not possible. Therefore, detailed morphology studies and measurement of the volume fraction of blocky retained austenite were performed using the EBSD method.

The study of the microstructure of nanobainitic steel with the use of a transmission electron microscope allows the identification of the following microstructure components: carbide-free bainite nanolaths, retained nanolathy austenite located between bainite laths, blocky retained austenite and precipitates – if thermodynamic conditions for their formation occurred in the manufacturing process.

TEM microstructure examination results are presented in Fig. 11. The observed type of lathy and blocky retained austenite morphology using the electron diffraction method and the dark field of view technique confirmed the presence of two austenite fractions in nanobainitic steel after the final heat treatment. No carbide precipitates were observed in the study.

4.3.2. Results of retained austenite examination

The results of retained austenite volume fraction measurement are presented in Table 7. Table 7 also presents the results of measurement of the volume fraction of blocky retained austenite determined using the EBSD method for the 5000× and 8000× magnifications. At a magnification of 8000×, the minimum size of the area (austenite phase) that can be analysed is between 100 and 200 nm. For the

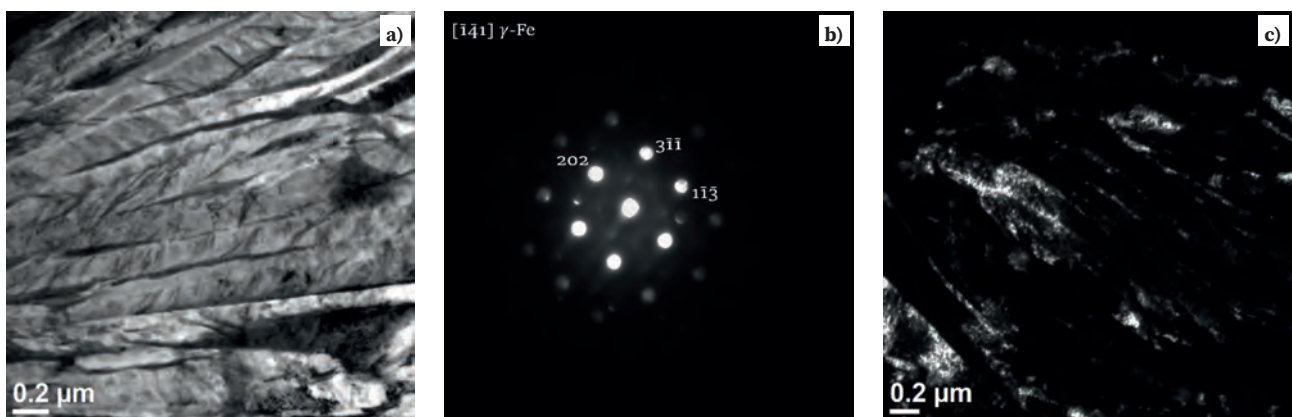


Fig. 11. Microstructure of nanobainitic steel, heat P1, variant 215/96; a) bright field image, b) electron diffraction pattern, c) dark field image in the reflex of retained lath austenite

Rys. 11. Mikrostruktura stali nanobainitycznej, wytop P1, wariant 215/96; a) jasne pole widzenia, b) dyfrakcja elektronowa, c) ciemne pole widzenia w refleksie austenitu resztkowego listwowego

Table 7. Results of retained austenite content measurement using the XRD and EBSD method**Tabela 7. Wyniki pomiarów zawartości austenitu resztkowego metodami XRD i EBSD**

Sample identification	Total retained austenite volume fraction, [vol%]	Retained austenite volume fraction, [vol%]	
	ξ_{XRD}	γ_{EBSD} magn. 5000 \times	γ_{EBSD} magn. 8000 \times
P1.210/96	14.5 \pm 1.6	11.1; 9.5	11.7; 11.4
P1.210/120	14.9 \pm 0.9	8.3; 10.5	8.3; 11.4
P1.215/96	14.8 \pm 2.2	9.6; 13.0	8.5; 15.1
P1.225/72	17.5 \pm 1.9	11.2; 12.3	12.1; 12.4
P2.210/48	22.5 \pm 2.7	7.8; 8.4	6.8; 8.6
P2.210/72	20.6 \pm 2.7	6.5; 5.5	6.7; 8.7
P2.210/96	15.5 \pm 1.5	10.9; 9.5	12.4; 9.5
P2.210/120	22.6 \pm 1.2	11.9; 9.9	13.4; 9.3
P2.215/96	25.0 \pm 1.2	10.4; 9.2	10.4; 8.2
P2.225/72	15.6 \pm 0.4	7.3; 6.4	9.2; 8.9

tested heat treatment variants from heat P1, the content of austenite was 15–17%. In the case of the P2 heat, the austenite content for the same heat treatment parameters as for P1 ranged from 20 to 25%. Similar contents of retained austenite were obtained by the authors of [8], which seems to be typical for nanobainitic steels and the isothermal transformation temperatures used. The volume fraction of retained austenite determined using the X-ray method is higher than the values determined using the EBSD method. This is confirmed by the presence of a nanolathy austenite fraction, which is not identifiable using the EBSD method.

The investigation of the morphology and volume fraction of blocky retained austenite was performed in SEM using

the EBSD method at three magnifications of 3000 \times , 5000 \times and 8000 \times . The smallest of the magnifications allowed for the analysis of the uniformity of blocky austenite distribution – e.g. in the areas of banded microstructure, but the minimum identifiable grain size of the austenite phase was limited to approx. 1 μm . Therefore, the content of retained austenite determined at the magnification of 3000 \times does not take into account the entire blocky austenite fraction. Blocky austenite grains/laths with sizes ranging from 100–200 nm (0.1–0.2 μm) were observed at 5000 \times and 8000 \times magnification. The measurement results of the fraction of blocky retained austenite volume at a magnification of 5000 \times were used for further analyses, e.g. to determine the content of nanolathy austenite.

Fig. 12 presents exemplary test results for blocky retained austenite in the P2 heat material after the final heat treatment. Studies at 3000 \times magnification showed the occurrence of inhomogeneity in the distribution of retained austenite with a grain size greater than 1 μm . This austenite fraction was characterised by a morphology close to equiaxed (globular) (Fig. 12 a,b). The distances between the zones with an increased content of blocky austenite (10–20 μm), the location of these zones and their width correspond to the banding of the microstructure of the plates found using the light microscopy examination. At the magnification of 5000 \times and 8000 \times , two types of retained austenite morphology were observed: globular grains and laths. The width of the laths and the mean linear intercept were in the range of 0.1–1.0 μm , and the maximum length of the laths reached approx. 3 μm (Fig. 12c–e).

4.3.3. Results of measurement of mechanical properties in static tensile test

The measurement of mechanical properties was taken on flat samples taken from hot-rolled plates, which were finally heat treated in heating furnaces at Łukasiewicz –

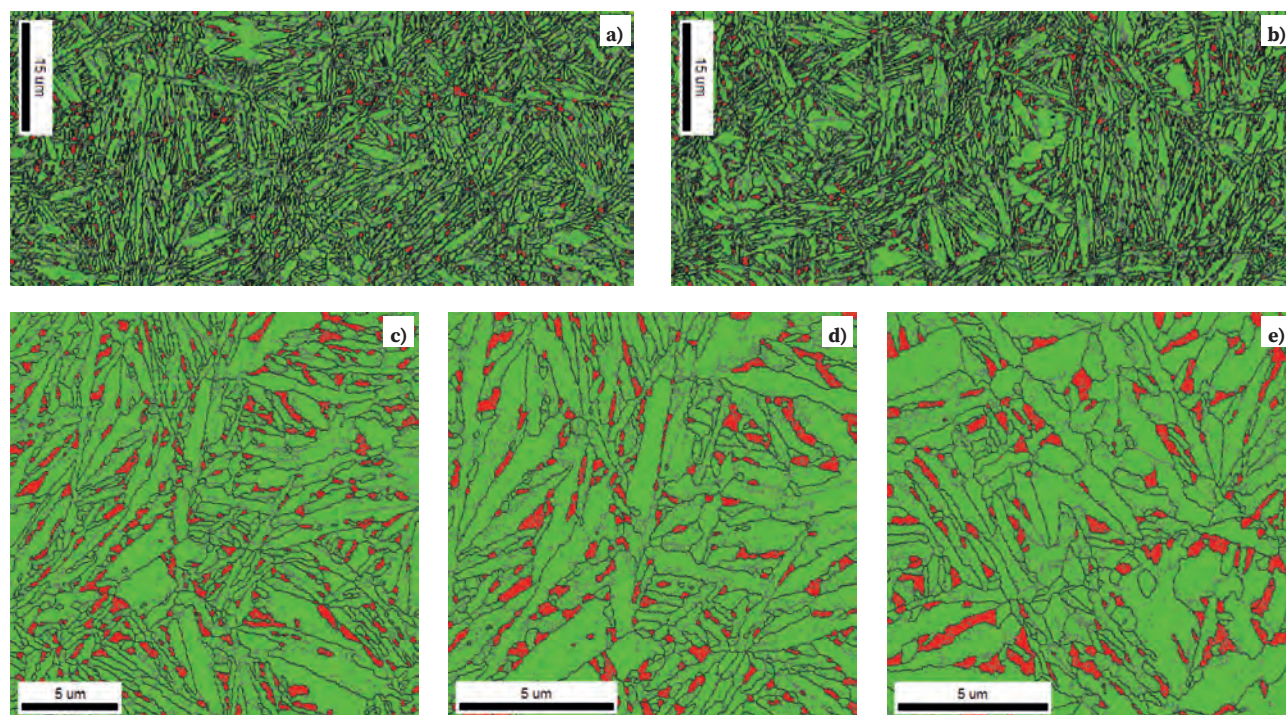


Fig. 12. Results of volume fraction measurement and blocky retained austenite morphology investigation. Heat P2, variant 210/96 (a, c, d) and variant 215/96 (b, e). a,b) magn. 3000 \times ; c) magn. 5000 \times ; d,e) magn. 8000 \times . Scanning electron microscope, EBSD method

Rys. 12. Wyniki pomiarów ułamka objętości oraz badań morfologii austenitu resztkowego blokowego. Wytop P2, wariant 210/96 (a, c, d) i wariant 215/96 (b, e). a,b) pow. 3000 \times ; c) pow. 5000 \times ; d,e) 8000 \times . Skanujący mikroskop elektronowy, metoda EBSD

IMŻ. The results of the measurement of the mechanical properties are presented in Tables 8 and 9, respectively, for the P1 and P2 heats, and in Table 10, the results of the impact strength measurement. In accordance with the assumptions of the project, the tensile strength was obtained for several variants of heat treatment and for both heats, with a total elongation greater than 12%. Nanobainitic steel is characterised by a wide range of deformation strengthening after exceeding the yield strength (YS) of 1300 to 1500 MPa for the following ranges of isothermal annealing parameters: temperature $T = 210, 215, 225^{\circ}\text{C}$; time $t = 120, 96, 72$ hours, respectively. The obtained results of the measurement of properties in the static tensile test are similar to the results of [3–10] in terms of strength, howev-

er, a higher elongation of 15% was obtained. It should be noted that most of the published results of studies on nanobainitic steels are conducted for a material with a higher carbon content, e.g. 0.9%, and a different content of alloying elements. Figs. 13 and 14 present typical tensile curves. The uniaxial compression tests were also carried out in the range of strain rate up to 100 s^{-1} . The test results are presented in Fig. 15. A stress level above 2500 MPa with a strain value up to 0.9 was obtained. During compression, the samples exhibited good ductility and a wide range of deformation hardening. The strength value is analogous to that obtained in [8] for steel with a higher carbon content and similar content of alloying elements.

Table 8. Results of the measurement of mechanical properties in a static tensile test, nanobainitic steel, heat P1

Tabela 8. Wyniki pomiarów właściwości mechanicznych w statycznej próbie rozciągania, stal nanobainityczna, wytop P1

Variant identification (950°C/30 min+ cooling in the air)	YS [MPa]	TS [MPa]	TE [%]	TS/YS
210/96	1349	2042	12.8	1.51
	1440	2045	14.4	1.42
	1376	2055	14.0	1.49
210/120	1525	2076	12.8	1.36
	1489	2067	14.4	1.39
	1510	2062	15.2	1.37
215/96	1531	2050	8.8	1.34
	1536	2048	12.6	1.33
	1453	2047	9.8	1.41
	1407	2087	11.8	1.48
	1460	2070	11.4	1.42
	1474	2072	11.6	1.41
225/72	1575	1974	14.0	1.25
	1355	1966	13.2	1.45
	1477	1961	14.2	1.33

Table 9. Results of measurement of mechanical properties in static tensile test. Nanobainitic steel, heat P2

Tabela 9. Wyniki pomiarów właściwości mechanicznych w statycznej próbie rozciągania. Stal nanobainityczna, wytop P2

Variant identification (950°C/30 min+ cooling in the air)	YS [MPa]	TS [MPa]	TE [%]	TS/YS
210/48	988	2199	13.2	2.23
210/72	1153	2158	12.0	1.87
210/96	1318	2040	14.0	1.55
	1318	2021	14.4	1.53
	1349	2035	15.4	1.51
210/120	1309	1989	12.2	1.52
	1425	1997	15.0	1.40
	1387	2000	14.2	1.44
215/96	1316	1972	14.0	1.50
	1328	1991	14.4	1.50
	1317	1987	15.4	1.51
	1371	2044	12.4	1.49
	1411	2044	12.9	1.45
	1399	2047	13.1	1.46
	1333	1959	15.0	1.47
225/72	1340	1936	14.0	1.44
	1357	1946	15.4	1.43

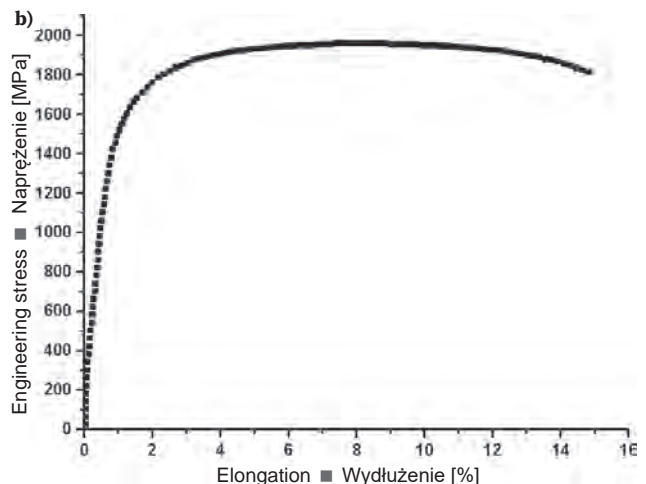
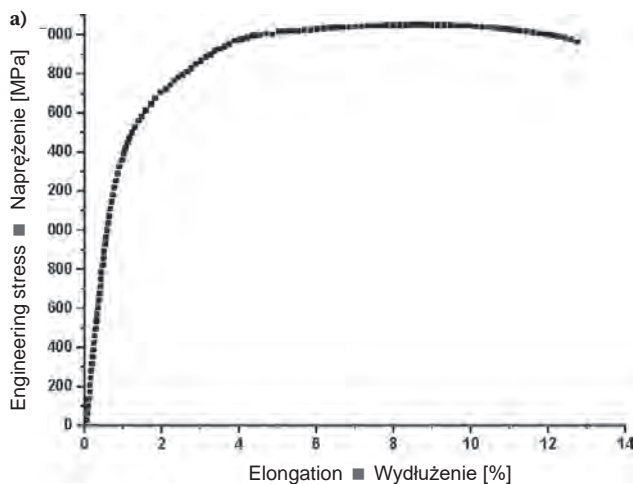


Fig. 13. Typical tensile curves for nanobainitic steel plate samples. Heat P1, a) 210/96; b) 225/72

Rys. 13. Przykładowe krzywe rozciągania próbek z blach ze stali nanobainitycznej. Wytop P1, a) 210/96; b) 225/72

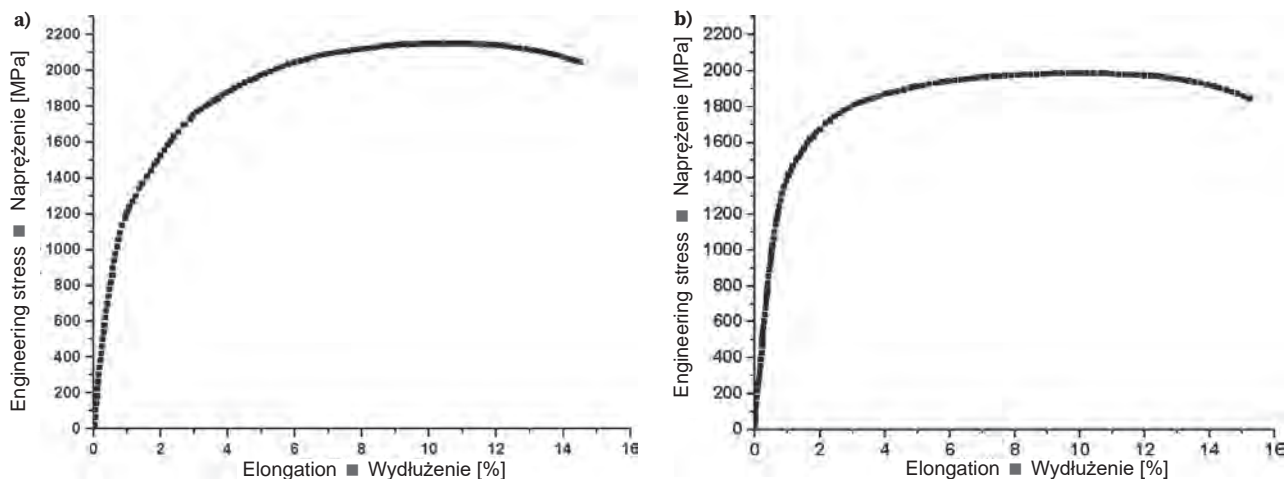


Fig. 14. Typical tensile curves for nanobainitic steel plate. Heat P2, a) 210/72; b) 215/96

Rys. 14. Przykładowe krzywe rozciągania próbek z blach ze stali nanobainitycznej. Wytop P2, a) 210/72; b) 215/96

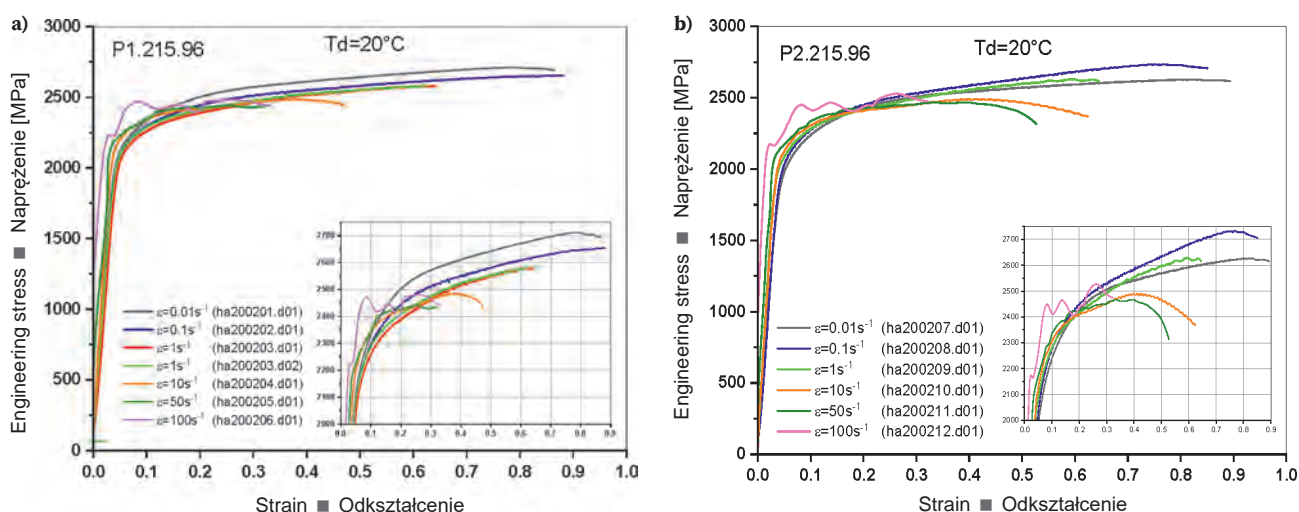


Fig. 15. Compression curves for nanobainitic steel within the strain rate in the range of 0.01–100 s⁻¹. Heat P1 (a) and P2 (b), isothermal annealing parameters 215°C/96 hours

Rys. 15. Krzywe ściskania stali nanobainitycznej w zakresie prędkości odkształcenia 0,01–100 s⁻¹. Wytop P1 (a) i P2 (b), parametry wygrzewania izotermicznego 215°C/96 godzin

Table 10. Charpy-V impact strength measurement results for nanostructured bainitic steel, samples with a cross-section of 10 × 7.5 mm. The values were calculated for a standard sample size of 10 × 10 mm

Tabela 10. Wyniki pomiarów udarności Charpy-V stali nanostrukturanej bainitycznej, próbki o przekroju 10 × 7,5 mm. Wartości przeliczono dla standardowego wymiaru próbki 10 × 10 mm

Identification of heat and HT variant temp. [°C] / time, [h]	-60°C [J]	-40°C [J]	-20°C [J]	0°C [J]	+20°C [J]	+40°C [J]
P1 225/72	8	11	12	13	19	21
P1 215/96	9	11	12	16	18	19
P1 210/96	10	12	14	15	19	20
P2 225/72	10	11	13	18	21	27
P2 215/96	9	10	13	17	21	22
P2 210/96	12	13	15	18	20	22

4.4. TWO-STAGE ISOTHERMAL TREATMENT

An alternative method of final heat treatment has been proposed, consisting in isothermal annealing at two tem-

peratures successively, which contributes to the improvement of the homogeneity of the properties distribution on the surface of the plate. The reason for the heterogeneity of the properties and microstructure of the plates is primary segregation and the resulting banding of the microstructure. The degree of primary segregation in industrial ingots varies between the “head” and the “foot” and as a result, the obtained plates may differ in quality in terms of material homogeneity. Examples of research and analysis results that were used to develop the heat treatment parameters in industrial conditions are presented below. On the basis of earlier tests, the first treatment temperature was selected at 225°C, and the second at 210°C. Times from 6 to 20 hours at 225°C and from 89 do 51 hours at 210°C were applied. The total transformation time, including the cooling time, as the temperature dropped from 225°C to 210°C, was either 72 or 96 hours. The temperature change from 225°C to 210°C was carried out in the furnace by reducing the set temperature during approx. 1 hour. Fig. 16 presents transformation kinetics curves for isothermal annealing variants in two temperatures. After heat treatment, the samples were subjected to microstructure examination

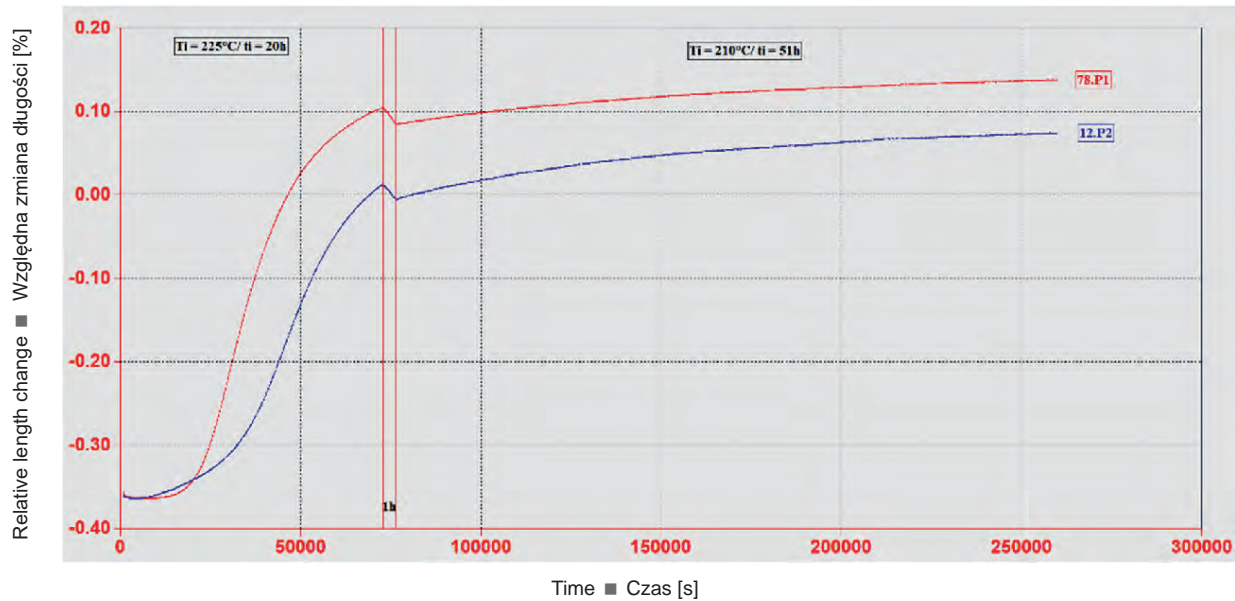


Fig. 16. Changes in the length of the dilatometric sample during annealing at 225°C for 20 hours and then at 210°C for 51 hours. The cooling time from 225 to 210°C was 1 hour. Heat P1 and P2

Rys. 16. Zmiany długości próbki dylatometrycznej w trakcie wygrzewania w temperaturze 225°C przez 20 godzin i następnie w 210°C przez 51 godzin. Czas chłodzenia od 225 do 210°C wynosił 1 godzinę. Wytopy P1 i P2

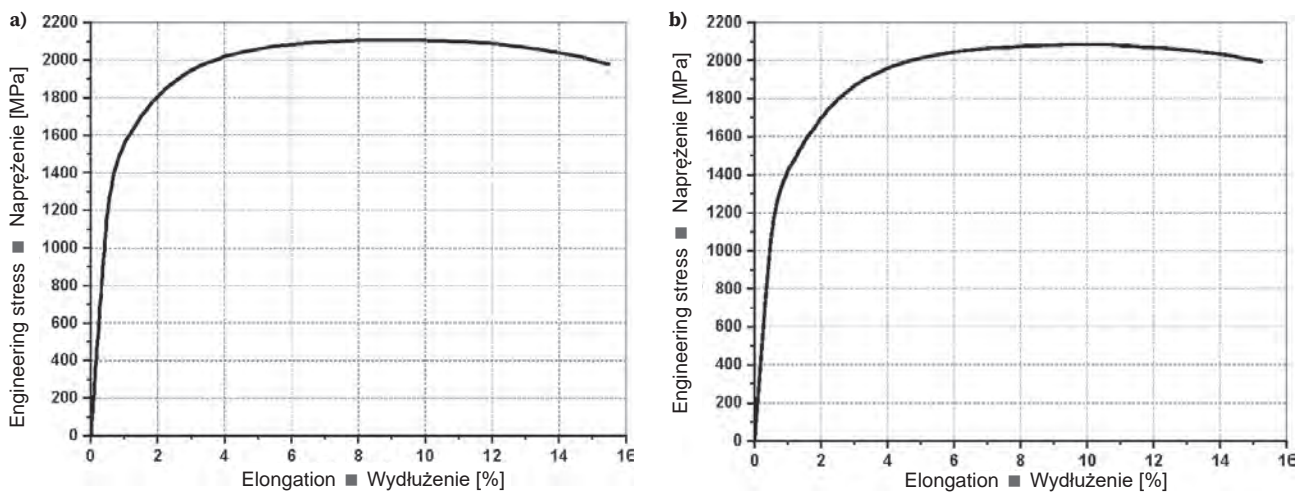


Fig. 17. Typical tensile curves for nanostructured bainitic steel plate. HT: 225/12+210/84; Heat: a) P1, b) P2

Rys. 17. Przykładowe krzywe rozciągania próbek z blach ze stali nanostrukturalnej bainitycznej. OC: 225/12+210/84; Wytopy: a) P1, b) P2

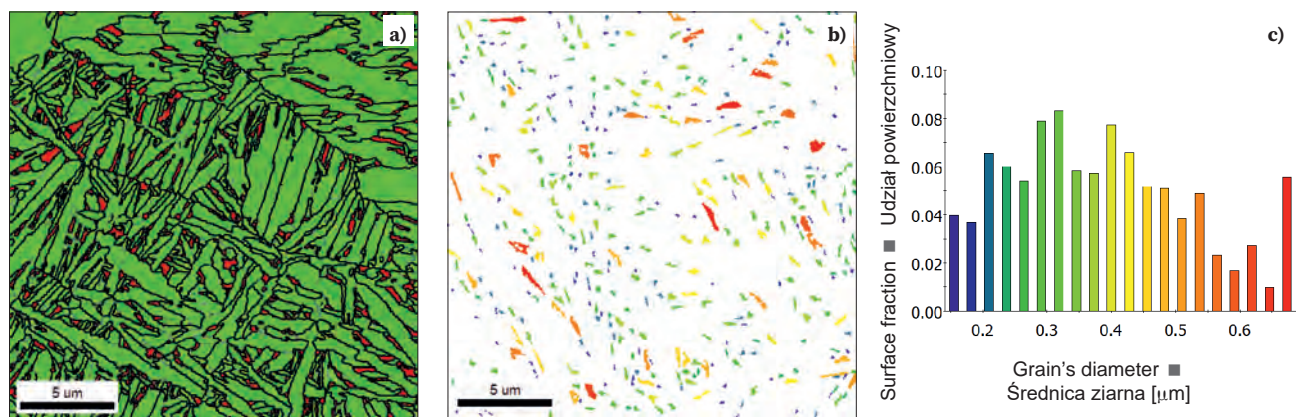


Fig. 18. Results of measurement of size distribution, volume fraction as well as morphology and distribution of blocks retained austenite. Heat P1, variant 225/12+210/84, magn. 5000×, scanning electron microscope, EBSD method. Number of grains 443, d_{av} 280 nm

Rys. 18. Wyniki pomiarów rozkładu wielkości, ułamka objętości oraz badań morfologii i rozmieszczenia austenitu resztkowego blokowego. Wytopy P1, wariant 225/12+210/84, pow. 5000×, skaningowy mikroskop elektronowy, metoda EBSD. Liczba ziaren 443, d_{sr} 280 nm

and hardness measurement. The results of the analysis showed the optimal variants of this type of treatment from the point of view of mechanical properties and resistance to perforation. The transformation time at 225°C was established to be 12 hours followed by 60 or 84 hours at 210°C. These variants were subjected to further microstructure and mechanical properties tests, and the plates were prepared for firing tests. The results of testing the mechanical properties of samples after isothermal annealing at two temperatures are presented in Table 11, and Table 12 presents the results of impact strength measurement. Figures 17 and 18 present typical results of the examination of properties and microstructure. The comprehensive analysis of the test results indicated the final parameters to be used in the technology of heat treatment of plates in industrial conditions.

Table 11. Results of measurement of mechanical properties in static tensile test T and t – temperature and isothermal annealing time, respectively; austenitising parameters: 950°C / 30 minutes

Tabela 11. Wyniki pomiarów właściwości mechanicznych w statycznej próbie rozciągania. T i t – odpowiednio temperatura i czas wygrzewania izotermicznego, parametry austenitizowania: 950°C / 30 minut

Variant identification T1.t1+T2.t2 T, [°C]; t, [h]	Sample thickness, [mm]	YS [MPa]	TS [MPa]	TE [%]	TS/YS
P1					
225.6+210.90	8.73	1350	2067	13.7	1.53
225.6+210.90	8.73	1333	2087	13.2	1.57
225.6+210.90	8.73	1378	2064	13.3	1.50
225.12+210.60	8.87	1395	2101	11.1	1.51
225.12+210.60	8.73	1290	2117	11.3	1.64
225.12+210.60	8.77	1320	2108	11.1	1.60
225.12+210.84	8.76	1451	2105	11.3	1.45
225.12+210.84	8.76	1453	2112	11.1	1.45
225.12+210.84	8.77	1469	2124	11.3	1.45
P2					
225.6+210.90	8.63	1280	2063	14.2	1.61
225.6+210.90	8.63	1291	2060	13.7	1.60
225.6+210.90	8.64	1320	2064	14.0	1.56
225.12+210.60	8.82	1230	2118	11.8	1.72
225.12+210.60	8.82	1223	2104	10.2	1.72
225.12+210.84	8.83	1320	2086	13.1	1.58
225.12+210.84	8.83	1358	2077	13.3	1.53

Table 12. Charpy-V impact strength measurement results for nanostructured bainitic steel, samples with a cross-section of 10 × 7.5 mm. The values were calculated for a standard sample size of 10 × 10 mm

Tabela 12. Wyniki pomiarów udarności Charpy-V stali nanostrukturalnej bainitycznej, próbki o przekroju 10 × 7,5 mm. Wartości przeliczono dla standardowego wymiaru próbki 10 × 10 mm

Identification of heat and HT variant temp. °C / time, h	+20°C [J]	-40°C [J]
P1 225.12+210.84	19	11
P2 225.12+210.84	22	14

4.5. FIRING TEST RESULTS FOR INDUSTRIAL MATERIAL AFTER HEAT TREATMENT IN LABORATORY FURNACES

The results of the firing tests were a key stage of the verification of the applied plate production technology parameters. In the field of final heat treatment, based on the results of microstructure tests, measurement of mechanical properties and the results of firing tests, the range of temperature and time of isothermal annealing was determined, after which plates made of nanobainitic steel with a thickness of 7.5–8.5 mm show resistance to perforation under the conditions of firing according to STANAG 4569 – level 2. In the first stage, the research material consisted of industrial scale plates, which were subjected to various parameters of isothermal annealing in laboratory conditions. Based on the results of firing tests with the first series, variant P1.215.96 was selected. The plate with a nominal thickness of 8.5 mm made of this material fulfilled the firing test with a 7.62×39 API BZ ammunition at an angle of 0°, in the entire velocity range specified in Stanag 4569 – level 2 (695–715 m/s) and additionally at velocities significantly higher than the upper limit according to the above-mentioned document, amounting to 757 and 775 m/s. For the second series of firing tests, P1 and P2 heat plates were prepared with a wider range of parameters of the final heat treatment. Very good protection properties were demonstrated for the P2 heat plates. The requirements for level 2 according to Stanag 4569 were met by plates with a nominal thickness of 7.5 mm in three heat treatment variants (210.96; 215.96 and 225.72). In addition, these plates exhibited resistance to perforation with a 7.62×39 mm shell (0° angle) for a velocity of 765 m/s. In the second series of tests, the positive result obtained in the first series was confirmed for 8.5 mm plate from the P1 heat in the 215.96 variant, and additionally for this material, a positive result was obtained for the 8.5 mm thick plate in the 210.96 variant.

500×500 mm plates from heat P2 were prepared for the third series of firing tests. Heat treatment with the use of a furnace and a fan cooling system was carried out for these plates. The use of a fan may have the effect of surface cooling of a thin layer of material, including scale, as a result of which the temperature of the centre of the plate is higher than that measured on the surface with an optical pyrometer. Then, there is a risk of the formation of upper bainite when the temperature of isothermal annealing is reached, which is particularly unfavourable if it separates at grain boundaries of former austenite. Disparity of the plates' properties on the surface was found. There was perforation in a specific zone of the plate, and in the second part the test results were positive, also for the highest possible velocity for the 7.62×39 mm API BZ ammunition. For example, in the area of one 500×500 mm plate, for the velocity of 746 m/s, the test was positive, and for the velocity of 696 m/s, it was negative. In the third series of tests, a positive test result was obtained by firing at a plate with a thickness of 6.6 mm P2.215.96 at an angle of 30° in accordance with the requirements for Stanag 4569 level 2. It was assumed that the cooling of the plates after austenitisation would be carried out in the air, the more so as the alloying content of P2 steel ensures sufficient hardenability (critical cooling rate) for the correct implementation of the plate manufacturing process. Fig. 19 presents the photographs of nanobainitic steel plates after firing.

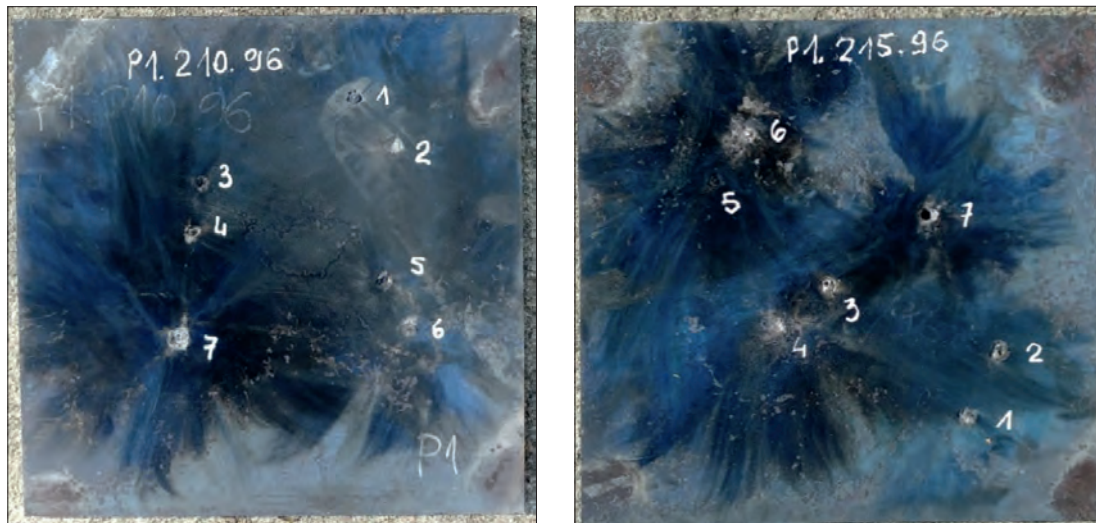


Fig. 19. Photographs of plates after firing with a 7.62×39 mm BZ ammunition, heat P1, thickness 8.5 mm

Rys. 19. Fotografie blach po ostrzale za pomocą amunicji kal. 7,62×39 mm BZ, wytop P1, grubość 8,5 mm

Results of microstructure examination within projectile impact areas

The study of the microstructure in the area of the projectile impact was performed in order to determine in detail the penetration depth and material characteristics in the

form of microcracks, shear bands, etc. Such tests provide additional information in terms of, for example, the margin of safety that a given material variant exhibits. Fig. 20 presents the results of macro- and microstructure examination within the projectile impact areas. Shear bands and

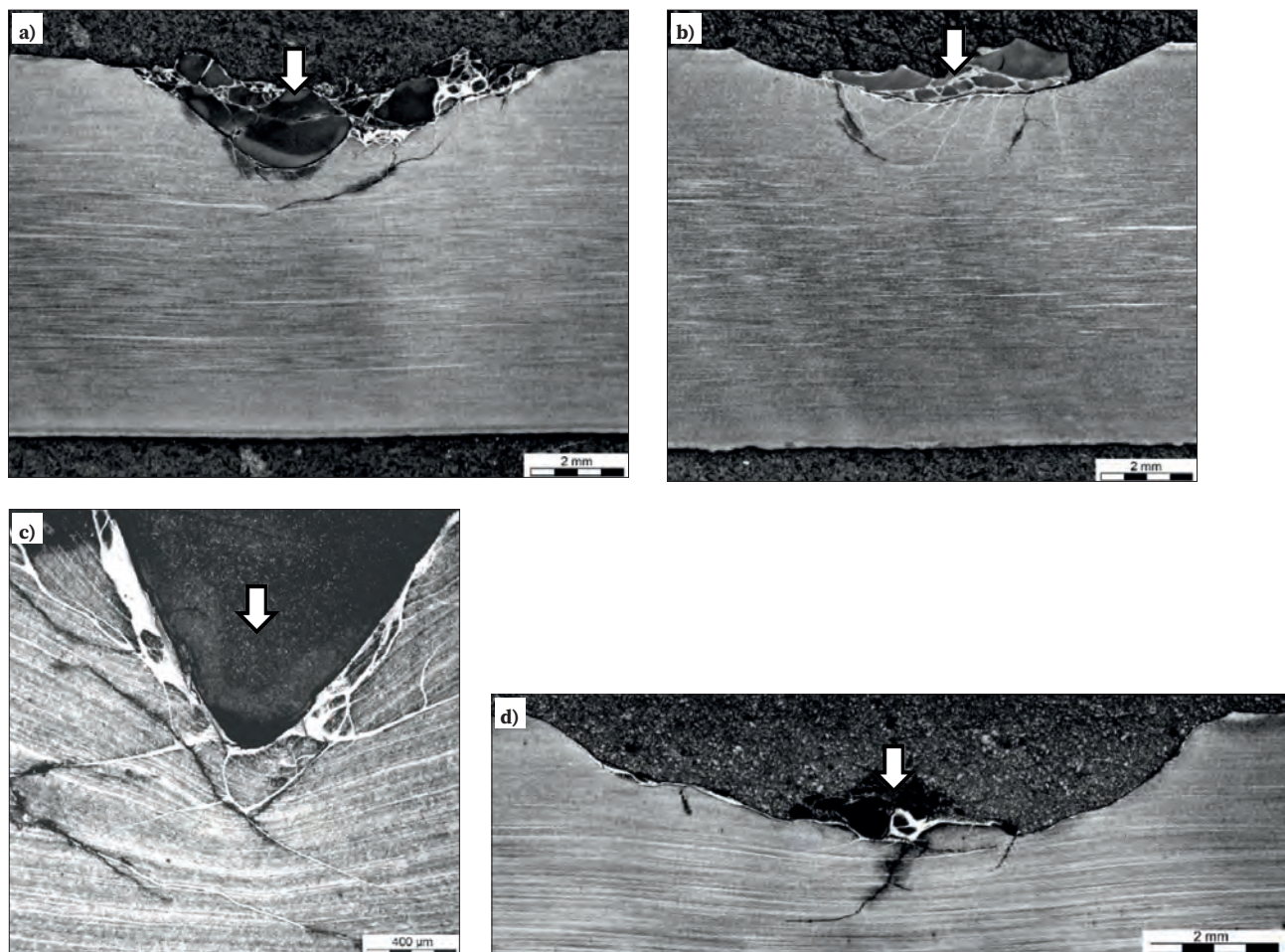


Fig. 20. Microstructure within the area of 7.62×39 mm API BZ projectile core impact on the plate; a) P2.225.12+210.60, plate thickness 8.3 mm; b) P1.225.12+210.84, plate thickness 9.0 mm, c) P1.210.96, plate thickness 8.5 mm, d) P2.210.96, plate thickness 7.5 mm

Rys. 20. Makrostruktura w obszarach uderzenia rdzenia pocisku 7,62×39 mm API BZ w blachę; a) P2.225.12+210.60, grubość blachy 8,3 mm, b) P1.225.12+210.84, grubość blachy 9,0 mm, c) P1.210.96, grubość blachy 8,5 mm, d) P2.210.96, grubość blachy 7,5 mm

cracks formed as a result of the firing were observed. There were also cracks with a characteristic curve in the direction parallel to the banding of the plate's microstructure. As part of the quality assessment, the hardness distribution in the thickness of the plates, which successfully passed the firing tests, was determined. The results of this measurement showed that in the subsurface zone with a depth of approx. 0.3 mm, the hardness decreased to approx. 500 HV, and in the remaining part the hardness was from 600 to 630 HV. Asymmetrical hardness distribution was observed along the plate's thickness. From one surface of the plates to a depth reaching half the thickness, the hardness was in the range of 600–610 HV, and on the other surface, the hardness was higher – in the range of 620–630 HV. The reason for this phenomenon could be one-sided decarburisation of the plate during the manufacturing process, e.g. during long-term soft annealing. The results of the effect assessment of the firing showed a positive reaction of nanobainitic steel to high-energy impact. The research showed the occurrence of various depths of penetration,

among others due to the typical heterogeneity of the material and the uniqueness of some parameters of the firing tests, including the alignment impact of the core, its shape and mass.

The test panels were subjected to ballistic tests. For the first series, panels were prepared that reflect the critical structural nodes of the container from the point of view of protective capacity. The armour of the panels consisted of nanobainitic steel plates from P1 and P2 heats, heat treated in the conditions of Łukasiewicz – IMŻ. As a result of the firing, the vast majority of tests were positive, including tests using a projectile velocity higher than the required one. There were single negative tests resulting from a local change in material properties caused by heterogeneity (primary segregation). For this purpose, additional firing tests were performed with the use of aramid laminate used as additional protection in the case when the base armour material meets the protective requirements, but there are single negative tests and/or borderline cases, e.g. in the form of secondary shrapnel. Table 13 presents firing

Table 13. Firing test results for experimental panels. Series 1

Tabela 13. Przykładowe wyniki testów ostrzałem paneli eksperymentalnych. Seria 1

Sample identification, material description	Test number	Projectile velocity V_U [m/s]	Result
P2 13C, P2 13D (8.0x500x500 mm)	1	711.1	No perforation
	2	722.3	No perforation
	3	691.8	No perforation
	4	723.5	No perforation
	5	717.7	No perforation
	6	719.8	No perforation
	7	750.1	No perforation
	8	714.3	No perforation
	9	704.9	No perforation
	10	717.4	No perforation
	11	705.1	No perforation
	12	704.8	No perforation
	13	713.7	No perforation

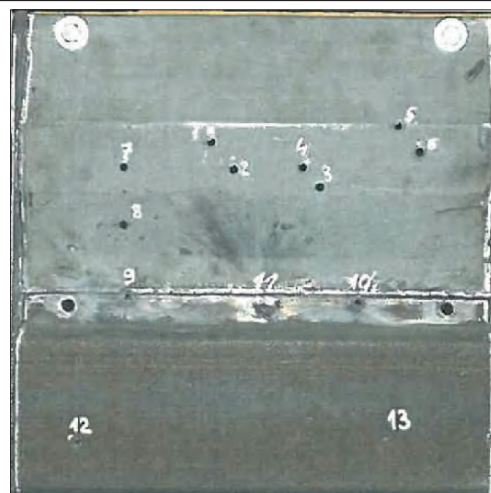
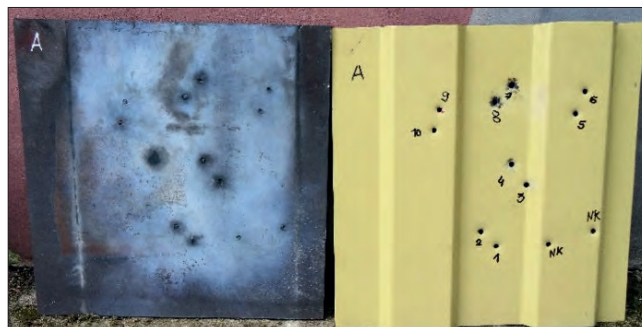


Table 14. Firing test results for experimental panels. Series 2

Tabela 14. Przykładowe wyniki testów ostrzałem paneli eksperymentalnych. Seria 2

Sample identification, material description	Test number	Projectile velocity V_U [m/s]	Result
P2: 225/12+210/60 (8.3x500x500 mm)	1	688.5	No perforation
	2	694.5	No perforation
	3	718.4	No perforation
	4	720.0	No perforation
	5	702.8	No perforation
	6	699.7	No perforation
	7	702.7	No perforation
	8	704.1	No perforation
	9	704.1	No perforation
	10	697.8	No perforation



test results and photographs of panels after testing. In the second series, the systems simulating the container walls with the use of armour made of nanobainitic plates, which were heat treated with modified parameters, were tested (Table 14). Contrary to the research conducted so far, a significant change was introduced in the final heat treatment technology and it was carried out at two temperatures with experimentally selected annealing time in each of them. The firing test results showed an improvement in protective parameters. In the vast majority of multi-hit firing tests, the requirements specified in Stanag 4569 for level 2 were met in 100%.

5. RESULTS OF RESEARCH ON THE MICROSTRUCTURE AND PROPERTIES OF NANOBAINITIC STEEL PLATES PRODUCED IN A FULL INDUSTRIAL PROCESS

The examination of the microstructure of test samples heat-treated in industrial conditions was carried out on the cross-sectional areas of the parts of the samples after tensile tests. Fig. 21 shows the microstructure images of the subsurface layer of the samples observed with a light microscope. Incomplete decarburisation to a depth of 0.12–0.15 mm was observed, which resulted in the formation of a different type of microstructure (lathy bainite and acicular ferrite) than nanobainite in this layer. Total decarburisation, which results in the formation of pre-ectoid ferrite, is in the form of single ferrite grains (Fig. 21a) or in places – a continuous thin ferrite layer with a thickness not exceeding 25–30 μm (Fig. 21b). Subsurface layer fragments not exhibiting complete decarburisation were also observed. When comparing the subsurface microstructure of the test samples with that of heat-treated plates with these samples, it should be taken into account that the surfaces of the samples were ground before heat treatment, while the surfaces of the plates were not cleaned or machined. The images of microstructure in the central area of the cross-section are presented in Fig. 22. Based on light microscopic observation of the microstructure of P1 and P2 steel samples heat-treated in an industrial process in sets together with the plates, the following conclusions can be drawn:

- The type of microstructure is correct for all tested variants - with slight deviations unavoidable in the industrial process, i.e. nanobainite is the matrix, and the remaining phase components are retained austenite in granular (block) form and – in some variants – traces of martensite lath (retained austenite less than approx. 0.5 μm cannot be identified using light microscopy).
- The microstructure is characterised by moderate banding, resulting from the segregation of alloying elements inherited at individual technological stages, formed during solidification of the steel; in light bands (with a higher concentration of elements) there is a higher content of retained austenite, and in darker bands (with a lower concentration of elements), in some variants, individual martensite laths were formed, due to the higher than the average M_s temperature for steel.
- There was no clear correlation between the microstructure features observed with a light microscope and the established differences in mechanical properties. This shows that the analysed small differences in the mechanical properties of the P1 and P2 steel plates presented in Table 15 depend on the parameters distinguishable in the nanoscale, such as the proportion of the amounts of individual morphological types of retained austenite and the dimensions and substructure of bainitic ferrite nanolaths as well as the form and distribution of carbon in retained austenite and bainitic ferrite.

For scanning electron microscope (SEM) studies, samples were selected that represent the morphological features of the microstructure in light microscopy studies. Images produced in SEM with the use of secondary electrons (SE - secondary electrons - images) show mainly the surface morphology – in this case, an etched relief derived from microstructure components – and therefore do not contain information on phase composition [19]. It was confirmed by comparative studies that with the SEM imaging method, also using the BSE – back scattered electrons - method, it is not possible to unequivocally identify areas of retained austenite. This shows that the grains of blocky retained austenite cannot be distinguished in the SEM images presented in Fig. 23. The SEM-SE images reveal the detailed structure of the steel matrix in the form of bainitic ferrite nanolath packets and sporadically occurring mar-

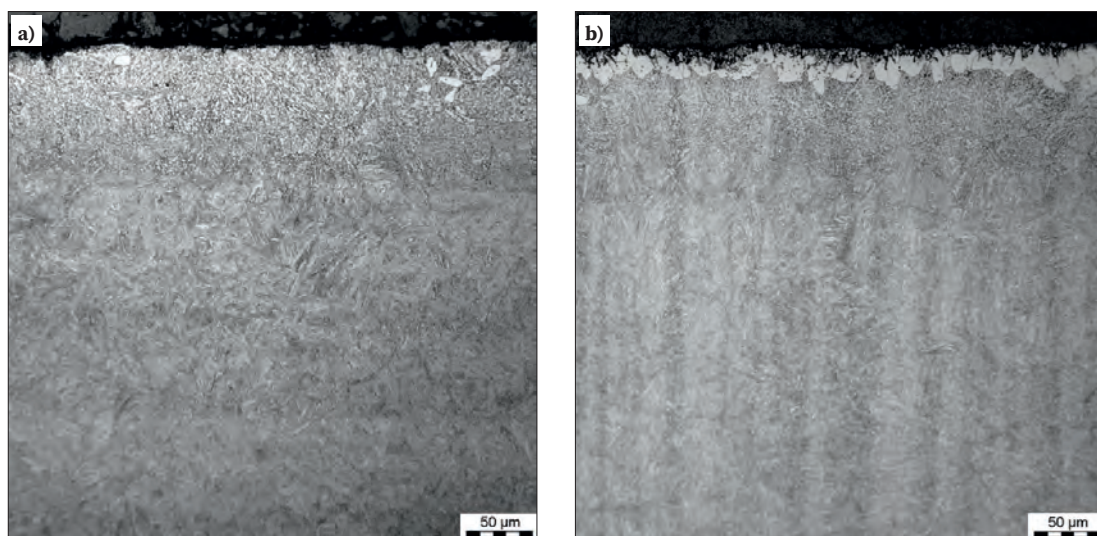


Fig. 21. Microstructure of the subsurface layer of heat-treated test samples with plate sets; a) P1 heat, b) P2 heat, light microscope

Rys. 21. Mikrostruktury przypowierzchniowej warstwy próbek testowych obrabianych cieplnie z pakietami blach; a) wytop P1, b) wytop P2, mikroskop świetlny

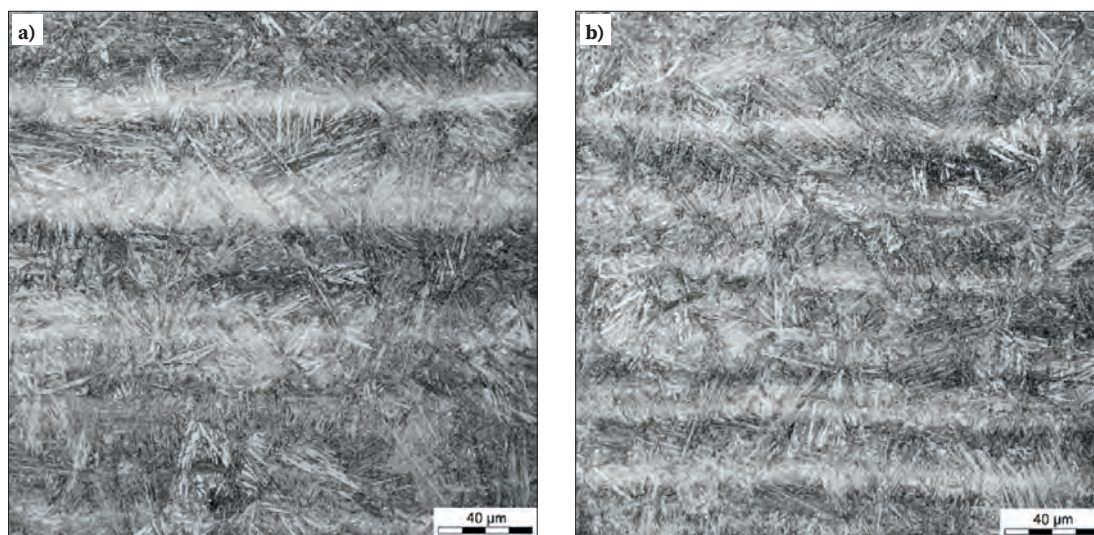


Fig. 22. Microstructures of central area in the cross-section of test samples, heat-treated with plate sets, P1 heat; light microscope

Rys. 22. Mikrostruktury środkowego obszaru na przekroju poprzecznym próbek testowych obrobionych cieplnie z pakietami blach, wytop P1; mikroskop świetlny

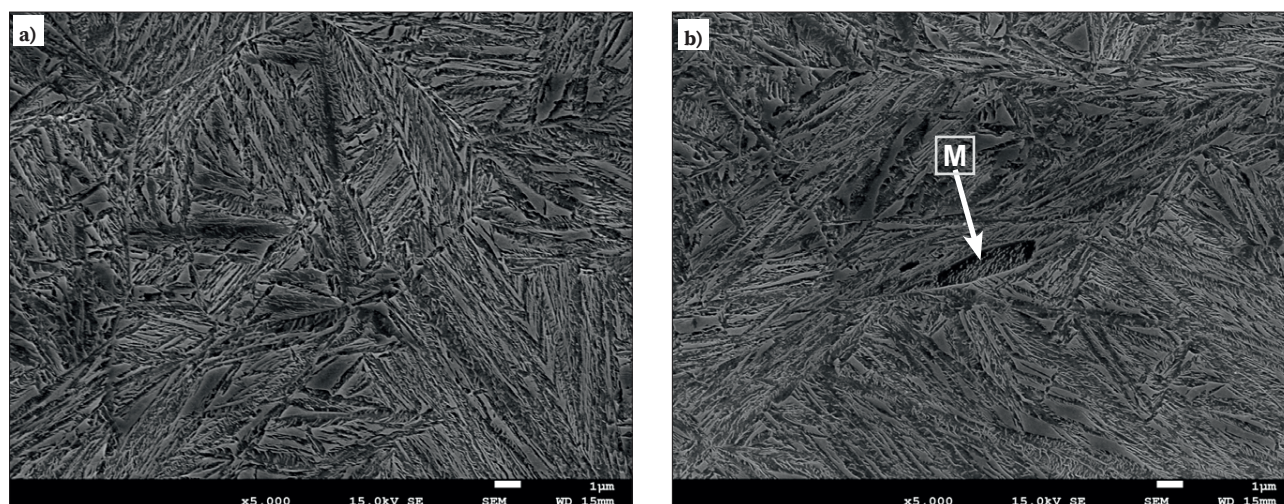


Fig. 23. Microstructures of central area in the cross-section of test samples, heat-treated with plate sets, scanning microscope, SEM-SE images; a) heat P2, b) heat P1

Rys. 23. Mikrostruktury środkowego obszaru na przekroju poprzecznym próbek testowych obrobionych cieplnie z pakietami blach, mikroskop skaningowy, obrazy SEM-SE; a) wytop P2, b) wytop P1

tensite laths (examples in Fig. 23b, marked as M). In some segregation areas with increased content of alloying elements, fine particles of undissolved carbides were found. In the tested samples, no high-temperature phase transformation products, such as upper bainite or pearlite, were found. The achieved microstructure type is analogous to the test results published in [6].

In order to verify that the microstructure of the test material (from the grip part of tensile samples) is representative of the microstructure of heat-treated plates in industrial conditions, samples were cut from P2 steel plates with a thickness of: 6.8 mm, 8.3 mm and 9.0 mm and tests were performed using a scanning electron microscope. Typical SEM images of the mentioned plate samples are presented in Fig. 24. The comparison of the microstructure of samples cut from plates with the microstructure of test strength samples heat treated together with the plates shows that the microstructure of tensile samples is made of identical bainitic ferrite nanolath packets as the

matrix of microstructure of plates randomly selected for the testing, and the number of individual martensite laths and areas containing undissolved carbide particles and the sizes of these particles are very close to the morphological parameters of the microstructure observed in the micrographs of strength samples. On this basis, it was assumed that the tensile samples represent the microstructure and mechanical properties of the plates processed with these samples in individual sets.

Fig. 25 shows the SEM images covering the edges of the P2 steel plate heat-treated in industrial conditions, illustrating the depth of decarburisation. Total (complete) decarburisation, resulting in the formation of pre-eutectoid ferrite grains, is small – it reaches a depth of approx. 50–60 μm and is approximately twice as large as the complete decarburisation of ground strength samples, which was assessed at 25–30 μm. The depth of incomplete decarburisation, reaching the point where the average hardness of the steel core is obtained, for a plate with a thickness

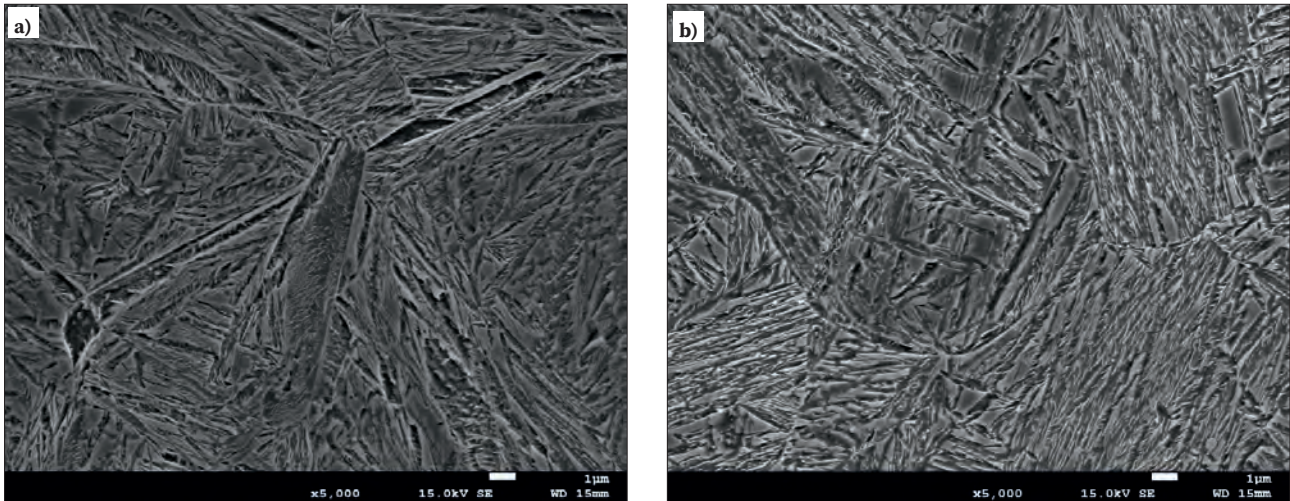


Fig. 24. Microstructures of central area on the cross-section of samples cut from P2 steel plates heat-treated under industrial conditions; a) plate thickness 6.8 mm; b) plate thickness 8.3 mm; scanning electron microscope

Rys. 24. Mikrostruktury środkowego obszaru na przekroju poprzecznym próbek wyciętych z blach ze stali P2 obrobionych cieplnie w warunkach przemysłowych; a) grubość blachy 6,8 mm; b) grubość blachy 8,3 mm; mikroskop skaningowy

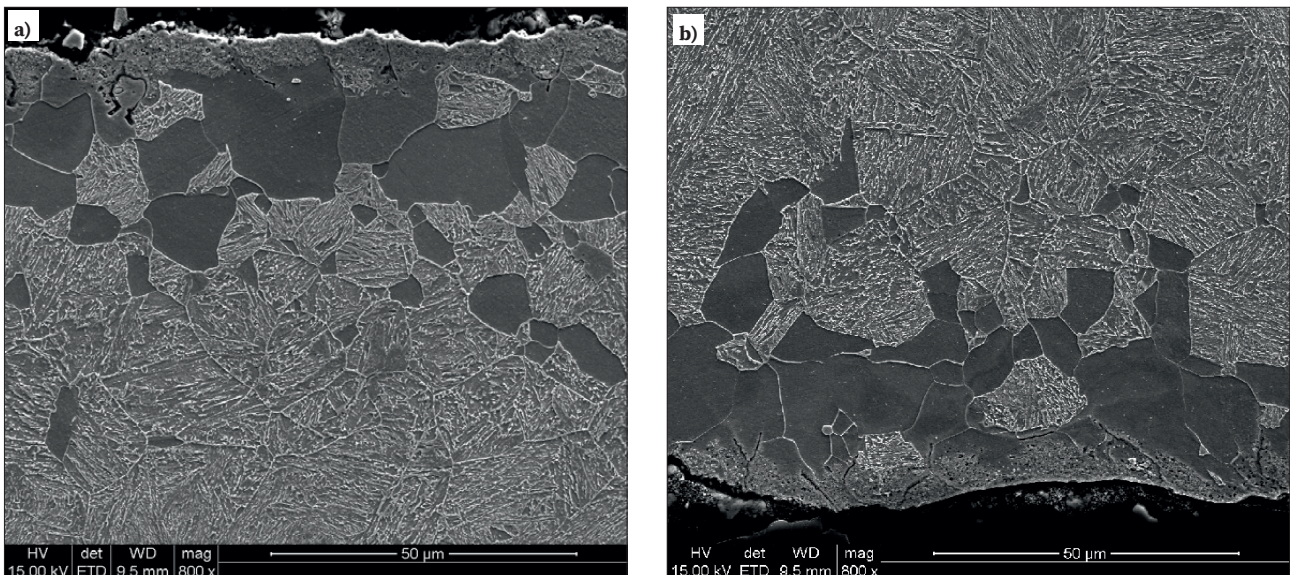


Fig. 25. SEM images covering the subsurface of the P2 steel plate heat-treated in industrial conditions

Rys. 25. Obrazy SEM obejmujące brzegi blachy ze stali P2 obrobionej cieplnie w warunkach przemysłowych

of 6.8 mm is approx. 0.5 mm. The microhardness diagram in Fig. 26 also shows the fluctuation of properties on the plate's cross-section resulting from the influence of element segregation. In the presented example, this causes the variability of the microhardness of the bands of approximately ± 10 HV in relation to the average value.

5.1. RESULTS OF EXAMINATION OF RETAINED AUSTENITE IN TEST SAMPLES AND PLATES HEAT-TREATED UNDER INDUSTRIAL CONDITIONS

The measurement of the total retained austenite (RA) content and the amount, morphology, size distribution and distribution of blocky retained austenite (bRA) after industrial heat treatment including a two-stage bainite transformation were performed on tensile samples (on the gripping part) suspended in sets of plates. The method of EBSD was used in a scanning electron microscope to determine the content and morphological parameters of bRA.

The measurement was taken at a 5000 \times magnification with the use of a single measurement area of 22.79 \times 51.81 μm . The smallest size of bRA grains detectable using the SEM-EBSD method was estimated at 0.15 μm . The segregation of elements, which was formed as a result of solidification and generally has the character of microsegregation, may appear in some places as a macrosegregation and has an influence on the heterogeneous distribution of RA grains and on the dispersion of the results of measurement of the content and morphology of RA.

The results of the measurement of the content, morphology and size distribution of retained austenite grains lead to the following conclusions:

- The average total RA content (measured using the XRD method) in the test samples made of steel P1 is 19.1 vol%, and in the test samples made of steel P2 it is 24.6 vol%. In the samples from P2 steel plates heat-treated together with the test samples, an average of 23.7 vol% of RA was obtained.

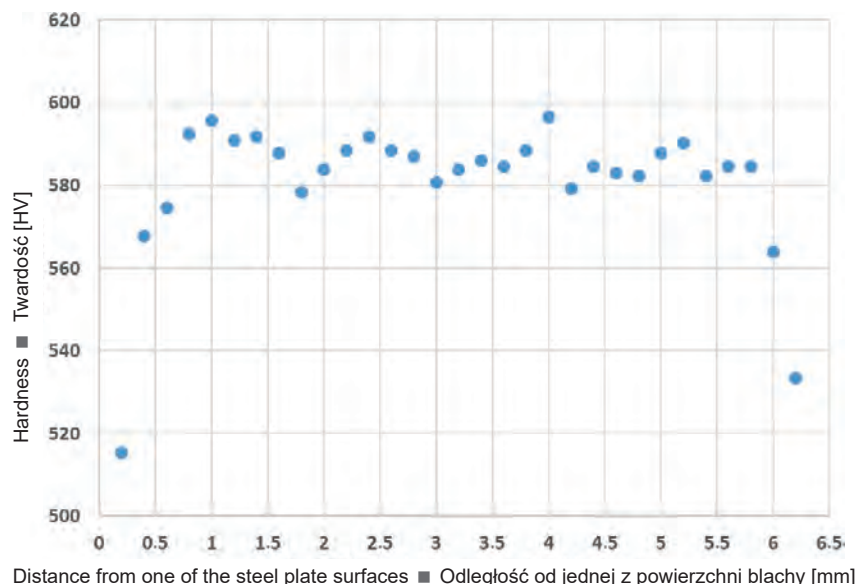


Fig. 26. Distribution of HV0.1 microhardness on the cross-section of 6.8 mm thick P2 steel plate heat-treated in industrial conditions

Rys. 26. Rozkład mikrotwardości HV0,1 na przekroju blachy o grubości 6,8 mm ze stali P2 obrobionej cieplnie w warunkach przemysłowych

- The average content of blocky retained austenite bRA with grains larger than $0.15\ \mu\text{m}$ (measured using the EBSD method) in the test samples of steel P1 is 5.2 vol% (in individual samples from 2.9% to 11.0%), and in test samples made of the P2 steel it is 3.5 vol% (in individual samples bRA 1.9% to 5.1%). In the samples cut from plates heat-treated together with the test samples, an average of 8.7 vol% of bRA (8.1 to 9.1%) was obtained.
- Based on the EBSD analysis, average and maximum bRA grain sizes were determined; for test samples made of steel P1, the average value is $0.39\ \mu\text{m}$ and the maximum value is $1.13\ \mu\text{m}$, for test samples made of steel P2 the average value is $0.41\ \mu\text{m}$ and the maximum value is $1.10\ \mu\text{m}$, and for samples cut from heat-treated plates together with test samples the average value is $0.57\ \mu\text{m}$ and the maximum value is $1.63\ \mu\text{m}$.

The given quantitative characteristics of retained austenite in nanobainitic steel after two-stage treatment differ statistically significantly from those after one-stage treatment.

5.2. ASSESSMENT OF THE EFFECTS OF TWO-STAGE TREATMENT

The mechanical properties of nanobainitic steel, in particular the resistance to impact, largely depend on the content, morphology, uniformity of distribution and dimensions of grains, lamellae and laths of retained austenite. Based on the laboratory tests carried out in this project, it was found that the final heat treatment leading to the production of a nanobainitic steel matrix, carried out in stages successively at two temperatures with a strictly defined holding time at each of these temperatures, leads to the formation of a microstructure containing blocky retained austenite with more favourable morphological parameters – a larger population of bRA grains with a smaller size and a smaller size of the largest regions of bRA was obtained. This regularity is illustrated by the comparison of the grain size distribution of bRA in P1 and P2 steel samples processed in laboratory conditions with the use of single-stage

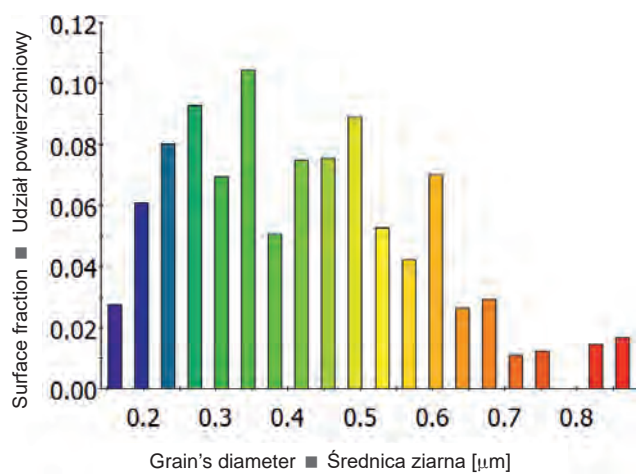
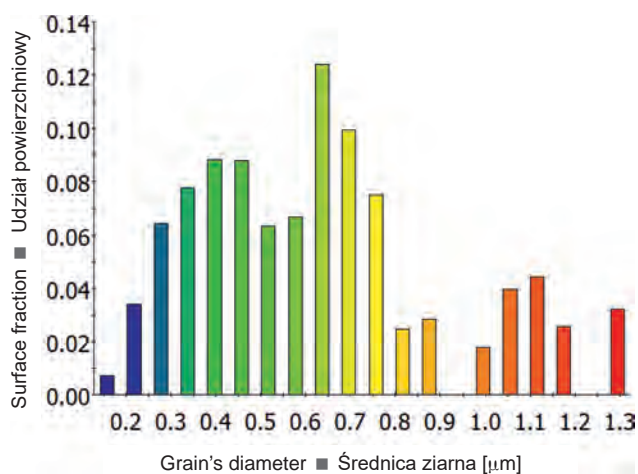


Fig. 27. Distribution of bRA grain sizes based on EBSD analysis in P1 steel samples after laboratory heat treatment: a) one-stage treatment: $210^\circ\text{C}/96\ \text{h}$, average bRA grain diameter $0.41\ \mu\text{m}$, b) two-stage treatment: $225^\circ\text{C}/12\ \text{h}+1\ \text{h}+210^\circ\text{C}/83\ \text{h}$, grain diameter of bRA $0.30\ \mu\text{m}$

Rys. 27. Rozkład wielkości ziarn ARb na podstawie analizy EBSD w próbkach ze stali P1 po laboratoryjnej obróbce cieplnej: a) obróbka jednoetapowa: $210^\circ\text{C}/96\ \text{godz.}$, średnia średnica ziarna ARb $0,41\ \mu\text{m}$, b) obróbka dwuetapowa: $225^\circ\text{C}/12\ \text{godz.}+1\ \text{godz.}+210^\circ\text{C}/83\ \text{godz.}$, średnia średnica ziarna ARb $0,30\ \mu\text{m}$

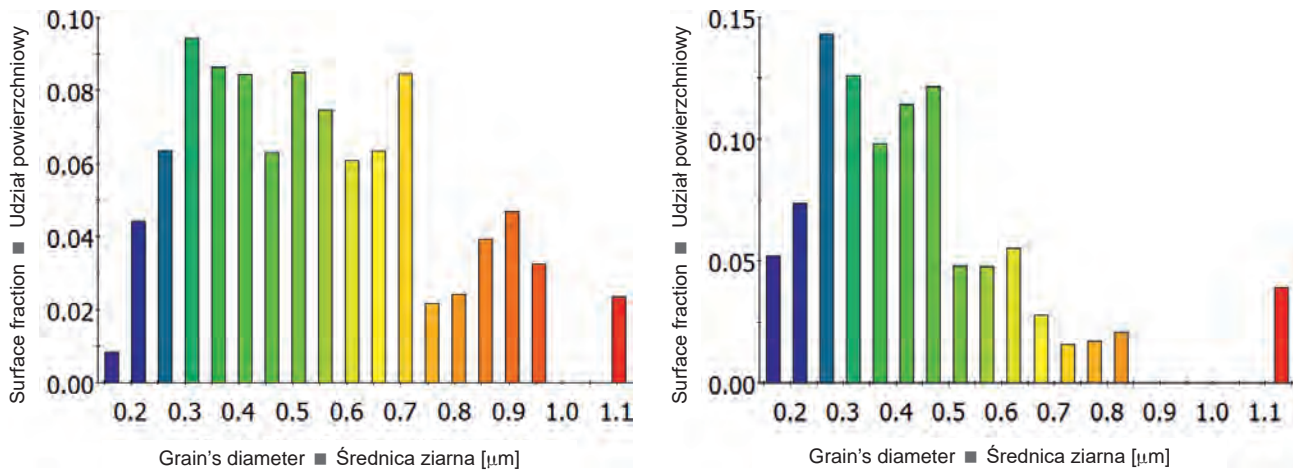


Fig. 28. Distribution of bRA grain sizes based on EBSD analysis in P2 steel samples after laboratory heat treatment: a) one-stage treatment at 210°C for 96 hours, average bRA grain diameter 0.37 μm, b) two-stage treatment 225°C/12 hours + 1 h + 210°C/83 hours, average bRA grain diameter 0.30 μm

Rys. 28. Rozkład wielkości ziarn ARb na podstawie analizy EBSD w próbkach ze stali P2 po laboratoryjnej obróbce cieplnej: a) obróbka jednoetapowa w 210°C w ciągu 96 godzin, średnia średnica ziarna ARb 0,37 μm, b) obróbka dwuetapowa 225°C/12 godz. + 1 godz. + 210°C/83 godz., średnia średnica ziarna ARb 0,30 μm

annealing (Figs. 27a and 28a) with two-stage annealing (Figs. 27b and 28b). An additional effect of the two-stage treatment is the increase in the total amount of RA in relation to the one-stage treatment, for P1 and P2 steels on average from 15–17 vol% to 21–24 vol%. As a result of the two-stage treatment in industrial conditions, the distribution of bRA grains with similar characteristics as in Figs. 27b and 28b was found, which confirms the adequate effect of the two-stage treatment on the grain refinement of the bRA phase.

5.3. MEASUREMENT RESULTS FOR MECHANICAL PROPERTIES OF HEAT-TREATED PLATES UNDER INDUSTRIAL CONDITIONS

Witness samples were attached to plates processed under industrial conditions. The results of the measurement of mechanical properties of witness samples are presented in Table 15. The required mechanical properties were achieved, which confirmed the correctness of the process. In the vast majority of cases, the assumed mechanical properties determined in the static tensile test were achieved. Single slight deviations from the requirements may result from the specific location of the samples in extremely unfavourable places of the plate set (e.g. in the corner area of the plate) and from a significantly smaller sample weight in relation to the plate's weight.

5.4. RESULTS OF IMPACT STRENGTH MEASUREMENT FOR SAMPLES AFTER TWO-STAGE HEAT TREATMENT

The results of impact strength measurement are presented in Table 16. The achieved impact energy values were converted to a standard sample width of 10 mm. The nanobainitic steel in the P1 variant, after treatment with 225/12+210/84, obtained the impact strength values of approx. 19 J and 11 J at +23 and -40°C, respectively. Slightly higher impact strength values were obtained for the P2 heat, which for the same heat treatment parameters amounted to approx. 21 J and 15 J, respectively at +23 and -40°C. The high level of impact strength, especially at -40°C - defined in the standards as a minimum of 12 J for a Char-

py-V sample, ensures the resistance of the tested steel grades to cracking under impact loads. Impact strength values similar to those achieved in the project are required for martensitic steels widely used in ballistic shields.

5.5. FIRING TEST RESULTS FOR PLATES PRODUCED IN A FULL PRODUCTION CYCLE UNDER INDUSTRIAL CONDITIONS

Firing tests were carried out for the plates' nominal thickness of 8.0 mm for the armour of the container's walls and for plates with a nominal thickness of 6.0 mm for the container's roof armour. The test result confirmed the achievement of the assumed protection level of the container's wall and roof models. Typical photographs of the plates after firing are presented in Fig. 29. The results of the firing tests showed the high potential of plates with the designed mechanical properties and thickness to meet the requirements of resistance to perforation in accordance with Stanag 4569 Level 2. In the mass plate production process, special attention should be paid to ensuring the uniformity of the chemical composition and properties on the surface and in the volume of a single plate. The results of firing tests indicated the necessity to strictly control the parameters at individual stages of production - from melting and casting, through processing, to the final heat treatment - in order to meet the protection requirements.

6. SUMMARY

Based on the tests of the material produced in laboratory conditions, the chemical composition of two grades of nanostructured bainitic steel was developed and guidelines for the following stages of industrial plate production: melting and casting, heat treatment of ingots before forging and slabs after forging, critical cooling rates and required temperature values during the process of rolling and immediately after rolling the plates. The material characteristics of industrial heats of two grades of nanobainitic steel were developed on the basis of dilatometric tests, measurement of mechanical properties and the volume

Table 15. Results of measurement of mechanical properties. Witness samples attached to plates during heat treatment in a test cycle

Tabela 15. Wyniki pomiarów właściwości mechanicznych. Próbki wytrzymałościowe „świadek” przymocowane do arkuszy blach w trakcie obróbki cieplnej w cyklu testowym

Sample identification	Location of sample in a plate set	YS [MPa]	TS [MPa]	TE [%]	UE* [%]	TS/YS	
Cycle 1	P1.1.1	Centre of plate 1 (first plate in the set from the furnace loading side)	1370	2070	10.0	6.1	1.52
	P2.1.1	Centre of plate 1 (first plate in the set from the furnace loading side)	1302	2050	11.2	8.7	1.56
	P1.1.2	Lower corner of plate 3 (third plate in the set from the furnace loading side)	1519	1997	12.2	8.7	1.32
	P2.1.2	Lower corner of plate 3 (third plate in the set from the furnace loading side)	1332	1973	13.8	6.7	1.52
	P1.1.3	Centre of plate 3 (third plate in the set from the furnace loading side)	1468	2067	13.4	8.0	1.41
	P2.1.3	Centre of plate 3 (third plate in the set from the furnace loading side)	1323	2048	14.6	10.0	1.54
	P1.1.4	Centre of plate 5 (central plate in the set)	1506	2086	13.0	8.4	1.39
	P2.1.4	Centre of plate 5 (central plate in the set)	1318	2053	12.0	9.1	1.56
Cycle 2	P1.2.1	Centre of plate 9 (last plate in the set from the furnace loading side)	1494	2075	12.2	7.7	1.39
	P2.2.1	Centre of plate 9 (last plate in the set from the furnace loading side)	1359	2063	12.4	9.7	1.52
	P1.2.5	Centre of plate 5 (central plate in the set)	1308	2048	11.4	7.5	1.56
	P2.2.5	Centre of plate 5 (central plate in the set)	1499	2040	13.4	8.4	1.37
Cycle 3	P1.3.1	Centre of plate 5 (central plate in the set)	1444	2034	11.8	8.2	1.41
	P2.3.1	Centre of plate 5 (central plate in the set)	1376	2011	12.6	8.8	1.47
	P1.3.2	Centre of plate 9 (last plate in the set from the furnace loading side)	1495	2028	10.4	6.6	1.35
	P2.3.2	Centre of plate 9 (last plate in the set from the furnace loading side)	1369	2007	11.6	7.5	1.47
Cycle 4	P1.4.1	Centre of plate 5 (central plate in the set)	1431	2080	14.0	9.0	1.45
	P2.4.1	Centre of plate 5 (central plate in the set)	1379	2060	12.6	9.9	1.49
	P1.4.2	Centre of plate 9 (last plate in the set from the furnace loading side)	1425	2051	13.0	8.5	1.45
	P2.4.2	Centre of plate 9 (last plate in the set from the furnace loading side)	1330	2040	12.8	8.5	1.54
Cycle 5	P1.5.1	Centre of plate 1 (first plate in the set from the furnace loading side)	1459	1992	12.0	8.2	1.37
	P2.5.1	Centre of plate 1 (first plate in the set from the furnace loading side)	1382	1968	13.1	9.2	1.43
	P1.5.2	Centre of plate 5 (central plate in the set)	1396	2002	12.0	7.1	1.43
	P2.5.2	Centre of plate 5 (central plate in the set)	1384	2008	11.8	7.2	1.45
	P1.5.3	Upper corner of plate 7 (seventh plate in the set from the furnace loading side)	1372	2034	12.9	8.7	1.49
	P2.5.3	Upper corner of plate 7 (seventh plate in the set from the furnace loading side)	1320	2016	13.1	8.9	1.54
	P1.5.4	Centre of plate 7 (seventh plate in the set from the furnace loading side)	1401	2008	13.1	8.7	1.43
	P2.5.4	Centre of plate 7 (seventh plate in the set from the furnace loading side)	1348	1993	13.8	9.0	1.47

*UE – uniform elongation

fraction of retained austenite, including microstructure examination in terms of the content of blocky and nanolath austenite. The main results of research and tests of plates made from the developed steel grades intended for

the armour of the observation and protective container are presented below.

- The following mechanical properties assumed to be achieved: $TS > 2.0$ GPa, $YS > 1.3$ GPa, $TE > 12\%$, were

Table 16. Results of Charpy-V impact strength (KV₂) measurement for material after two-stage heat treatmentTabela 16. Wyniki pomiarów uderności Charpy-V (KV₂) materiału po dwuetapowej obróbce cieplnej

Variant identification/ sample No.	Sample width [mm]	Sample height [mm]	Height below notch [mm]	Test temp. T, [°C]	Impact energy [J]	Impact energy converted per sample 10×10×55 mm [J]
P1.225.12_210.84/1	7.47	9.93	(7.93)	23	14	19
/2	7.45	9.95	(7.94)	23	14	19
/3	7.42	9.94	(8.07)	23	15	20
/4	7.47	9.93	(7.94)	-40	8	11
/5	7.45	9.95	(7.93)	-40	8	11
/6	7.46	9.94	(7.94)	-40	10	13
P2.225.12_210.84/1	7.47	9.93	(7.87)	23	16	21
/2	7.45	9.95	(7.96)	23	18	24
/3	7.46	9.94	(7.93)	23	16	21
/4	7.47	9.93	(7.93)	-40	9	12
/5	7.45	9.95	(8.07)	-40	11	15
/6	7.46	9.94	(8.06)	-40	11	15

met (or reached the level very close to the required values) by 79% of the test samples, while a few samples, to a relatively small extent, did not meet the established minimum levels of mechanical performance. No case was found that would disqualify the material in terms of the level of mechanical properties. This result should be considered very good for the first batches of plates heat-treated under industrial conditions. The average values of parameters determined in a tensile test for all test samples are:

- for steel P1: $TS = 2041$ MPa, $YS = 1435$ MPa, $TE = 12.2\%$,
- for steel P2: $TS = 2024$ MPa, $YS = 1359$ MPa, $TE = 12.8\%$.
- The correct type of plate's microstructure was obtained for all tested variants of heat treatment – with small deviations unavoidable in the industrial process, i.e. the matrix is nanobainite, and the remaining phase components are retained austenite in blocky form and – in some variants – traces of plate or lath martensite. Attention is paid to the influence of primary segregation and the resulting microstructure banding, which at a certain intensity (difference in the content of key elements, i.e.

Mo, Mn, Si and Cr in adjacent bands) can significantly change the functional properties of the material locally.

- The final treatment leading to the production of a nanobainitic steel matrix, carried out successively at two temperatures with a strictly defined holding time at each of these temperatures (225/12+210/84), leads to the formation of a microstructure containing blocky retained austenite with favourable morphological parameters – a large population of bRA grains with a smaller size and a smaller size of the largest regions of bRA was obtained. An additional effect of the two-stage treatment is the increase in the total volume fraction of RA in relation to the one-stage treatment, for P1 and P2 steels on average from 15–17 vol% to 21–24 vol%. The arrangement, morphology and size distribution of grains and laths of retained austenite are strongly dependent on the microsegregation of elements. The occurrence of segregation areas, especially with increased Mo content, was found, in which the estimated content and size of blocky austenite grains were larger compared to the areas of bands depleted in alloying elements.



Fig. 29. Plates after firing. a) heat P2, thickness 8.0 mm; b) heat P1, thickness 7.0 mm

Rys. 29. Fotografia blach po ostrzale. a) wytop P2, grubość 8,0 mm; b) wytop P1, grubość 7,0 mm

- Nanobainitic steel – P1 heat, after treatment 225/12 + 210/84, obtained impact strength values of approx. 19 J and 11 J at +23 and -40°C, respectively. Higher impact strength values were obtained for the P2 heat steel, which for the same HT parameters amounted to approx. 21 J and 15 J, respectively at +23 and -40°C. Similar impact strength values are required for quenched and tempered martensitic steels widely used in ballistic shields.

The results of the firing tests showed the high potential of plates with the designed mechanical properties and

thickness to meet the requirements of resistance to perforation in accordance with Stanag 4569 Level 2. In the mass plate production process, special attention should be paid to ensuring the uniformity of the chemical composition and properties on the surface and in the volume of a single plate. The results of firing tests indicated the necessity to strictly control the parameters at individual stages of production – from melting and casting, through processing, to the final heat treatment – in order to meet the requirements for ballistic protection.



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