ARCHIVES
of
FOUNDRY ENGINEERING
10.24425/afe.2025.155379
Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences
ISSN (2299-2944)
Volume 2025
Issue 4/2025
46 – 53
7/4

The Influence of Vanadium and Niobium Micro-additions on the Microstructure and Mechanical Properties of ADI

A. Zaczyński [a], *, M. Królikowski [a], A. Nowak [a], M. Sokolnicki [a], J. Jaroszek [a], A. Burbelko [b]
[a] Odlewnie Polskie S.A.,
27-200 Starachowice, inż. Władysława Rogowskiego Street 22, Poland
[b] AGH University of Krakow, Faculty of Foundry Engineering,
23 Reymonta Str., 30-059 Krakow, Poland
* Corresponding author: E-mail address: artur.zaczynski@odlewniepolskie.pl

Received 14.11.24; accepted in revised form 09.06.25; available online 22.12.2025

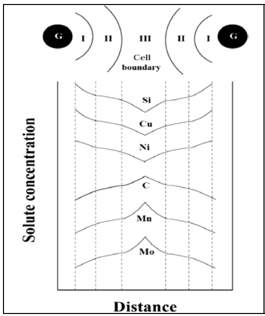
Abstract

Austempered Ductile Iron (ADI) casting technology is a combination of the smelting process, its post-furnace treatment and the heat treatment of castings. Maintaining the process parameter stability of this innovative high quality cast iron with high Tensile Strength UTS and ductility properties is the aim of a number of studies on the control of graphitization inoculation and inoculation of the metal matrix. The ability to graphitise the liquid alloy decreases with its holding in the furnace, time of pouring into moulds from pouring machines. The tendency to dendritic grains crystallization and the segregation of elements such as Si, Ni and Cu decrease the ductile properties. The austenitizing process can introduce austenite grains growth negatively affecting of the ausferrite morphology. The modifying effect of small amounts of additives on the metal matrix in steel and low alloy cast steel, well known in materials engineering, has been applied to ADI. The addition of cast iron chips, Fe-V and Fe-Nb to the liquid alloy in the first inoculation is an example of a hybrid interaction. The introduction of graphitization nucleus particles and austenite crystallisation nucleus particles resulted in a stabilisation of the ductility of ADI and an increase in mechanical properties. Grains refinement of the primary austenite and precipitation hardening of ausferrite stabilise the mechanical properties of ADI. As a result of graphitization and additive structure inoculation, graphite and ausferrite morphology is improved. The obtained results point the way to further research in the field of hybrid inoculation of Ductile Cast Iron.

Keywords: ADI, Austempering, Graphitization inoculation, Metal matrix inoculation, Hybrid inoculation

1. Introduction

Ausferritic ductile iron castings are assessed according to the criteria of mechanical properties contained in the EN 1564 standard. This cast iron is more and more often chosen by designers of machine and equipment parts as a competitive casting material. The conversion of ADI, in place of the previously used alloys, results in a reduction in the weight of the structure by up to several dozen percent. The assessment of mechanical properties is performed on the basis of samples obtained from test ingots or from the walls of castings indicated by the constructor (according to EN 1564). These are the zones with the highest operating stress values. To meet these requirements, technological processes (metallurgical-foundry and heat treatment) are designed to obtain in a stable technological margin of properties such as R_m, R_{p02}, A, HBW in relation to the requirements for a given cast iron grade and casting wall thickness. The chemical composition, parameters of post-furnace treatment and heat treatment of various grades of unalloyed cast iron [1] and alloyed cast iron [2] have been refined. In an intensive production process, the processes of cast iron melting, furnace pouring, magnesium treatment, inoculation and pouring of liquid alloy into casting moulds can be extended in time (up to 1.5 h for pouring and up to 12 minutes for pouring into moulds). The changing metallurgical quality of the liquid alloy during this period (the ability to grow graphite in the form of nodules) may result in the degeneration of the shape of graphite precipitates. The reasons for the deterioration of graphite morphology in the casting, resulting in a decrease in mechanical properties, are the segregation of elements [3] (Fig. 1), the dendritic microstructure of austenite, crystallization deviating from the optimal eutectic.



◆ FTF region (First to Freeze) or region I is eutectic cells near graphite nodules. Elements like Ni, Cu and Si with reverse segregation segregate in this region.
◆ The region with nearly constant composition or region II.
◆ LTF region (Last to Freeze) or region III is eutectic cells boundaries far from graphite nodules. Elements like Mo, Mn and Cr with positive segregation segregate in this region.

Fig. 1. Segregation of elements in cast iron [3]

Own experimental results, which will be presented below, indicate that particularly negative effects of process stability are associated with the elongation of A. Attempts to improve ductility with heat treatment parameters reduce the strength properties to the minimum limits. The instability of the cast iron structure applies especially to castings with a wall thickness of over 30 mm, mainly in the area of heat substations (risers). The aim of this research is to search for methods to stabilize the technological process of this high-strength casting material, classified as High Technology. One of the effective methods used in low-alloy steels [4-6] and cast steel [7] is the introduction of modifying additives of vanadium, niobium and titanium into the chemical composition of these materials. These additives, forming carbonitrides during production processes (crystallization, rolling, heat treatment), grind the original grain of austenite, as well as ferrite or bainite. The grinding of the grains had a positive effect on both the strength and ductility of the cast iron. Micro-additives are particularly widely used in nitrogen-enriched low-carbon manganese rolled steels. The formed primary nitrides only partially dissolve in austenite at rolling temperature (above 1050ºC). During cooling of the sheets after rolling (550 – 650 ºC), the release of new nitride particles of these elements leads to precipitation hardening, significantly increasing the values of Rp_{02} and R_m. The total effect of the increase in yield strength σ_y of ferritic micro-alloy steel was estimated according to the relation [4,5]:

$$\sigma y = \sigma_o + \sigma_s + \sigma_g + \sigma_{p+\sigma d},$$

where:

σ_o = 45 MPa – hardening of ferrite by dissolved C and N,

σ_s = 84(Si) + 32(Mn)+38(Cu)+43(Ni) MPa – strengthening of the matrix with the elements present,

$\sigma_g = 18.1\ d^{-1/2}$ MPa – hardening by grinding of ferrite grain with diameter d,

$\sigma_p = 10.8 \cdot f \cdot 1/2/d \cdot \ln(d/(6.125 \cdot 10^{-4}))$ MPa – precipitation reinforcement,

f – volume fraction of the precipitates,

d – average diameter of precipitates in microns; for $f \sim 13 \cdot 10^{-4}$, d = 6.5 nm, σ_p = 141 MPa,

$\sigma_d = 16{,}2 \cdot 10^{-4} \cdot \rho^{1/2}$ MPa - reinforcement by dislocations;

ρ is the dislocation density in cm^{-2}; for $\rho \sim 109$ lines·cm·cm^{-2}, σ_d = 51 MPa, for $\rho \sim 1010$ lines·cm·cm^{-2}, σ_d = 162 MPa

The effect of precipitation hardening and dislocation hardening of wrought steel was also estimated using the following relation [4]:

$$\sigma p + \sigma d = \sigma_y - (\sigma_o + \sigma_s + \sigma_g)$$

The introduction of micro-additives into ferritic ductile iron may also have a positive effect on the morphology of graphite, the number of carbide precipitates and the mechanical properties of cast iron [8, 9]. The addition of vanadium to the casting to the level of 0.08% resulted in an increase of the R_m value by 8%, R_{p02} by 13%, and the number of graphite precipitates by 10% with the unchanged elongation value (approx. 20%). With a vanadium content of 0.15%, a further strengthening of cast iron was observed with a decrease in the elongation value (17%). The influence of niobium was slightly weaker in the strengthening of cast iron [8].

Attempts to supplement the chemical composition of ausferritic cast iron with additives V and Nb have been presented in publications [10-12]. The presence of 0.35 wt.% Nb in cast iron subjected to austenitizing at 900ºC and austempering (320ºC and 360ºC) did not cause significant changes in mechanical properties compared to cast iron without niobium [11]. However, NbC precipitates were observed within the graphite "spheres". Considering that the crystallization of this carbide starts earlier than that of graphite, this may mean that NbC particles can be used as pads to nucleate graphite precipitates (Fig. 2).

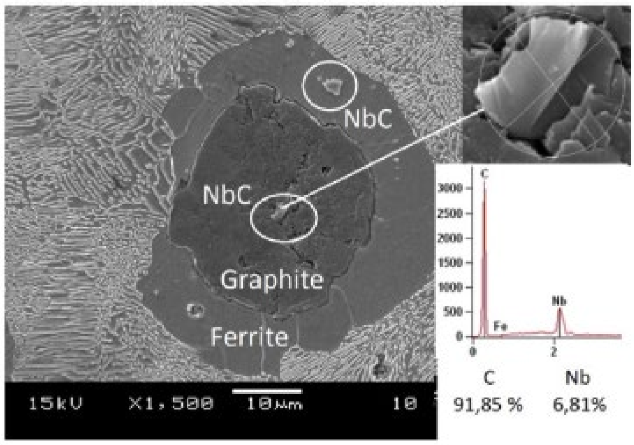


Fig. 2. NbC precipitation inside spheroidal graphite [11]

The influence of vanadium on the strength properties of cast iron is significant, both in the cast state and after heat treatment: austenitizing at 900°C and isothermal hardening at 265 and 305°C (Fig. 3) [12].

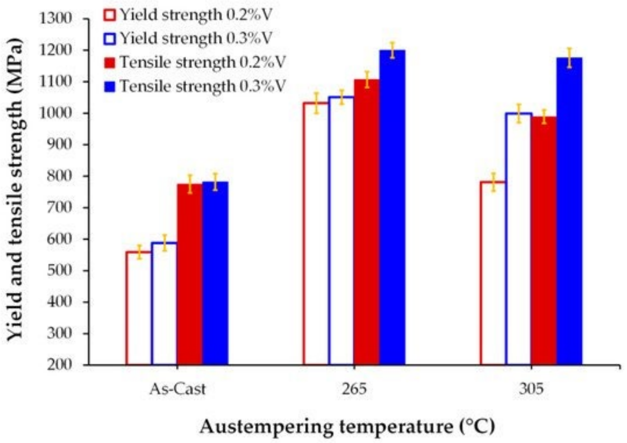


Fig. 3. The influence of vanadium on the value of the R_{p02} and R_m properties of ADI [12]

A significant strengthening effect of the material was found after austempering at a temperature of 305°C.

Analysing the presented research, a significant improvement in the strength properties of ADI with the addition of vanadium was found compared to niobium. At the austenitizing temperature of ADI (approx. 900°C), vanadium carbides dissolve more easily compared to Nb carbides (Fig. 4) [13].

As in rolled steels, the following processes may occur during the heat treatment of ADI:

- dissolution of primary micro-additive carbides in austenite during austenitizing; some undissolved carbide precipitates may prevent austenite grains growth,
- delayed precipitation of micro-additive-containing particles from austenite during rapid cooling of the alloy in salt baths,
- precipitation of highly dispersed carbides from the metal matrix during isothermal holding of the treated alloy in a salt bath during the austempering procedure.

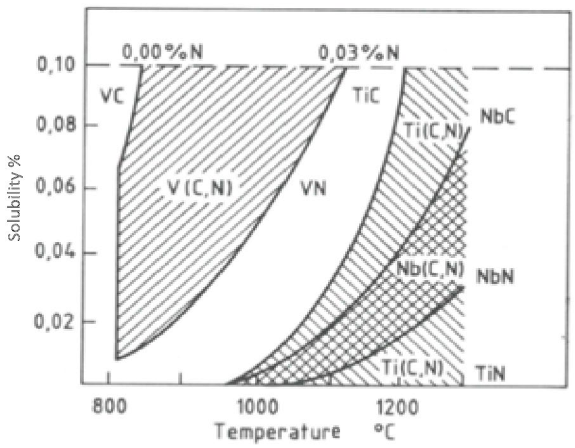


Fig. 4. Solubility of carbonitride micro-additions in austenite [13]

The introduction of vanadium and niobium micro-additions to the liquid alloy may affect the crystallization of cast iron, modifying the course of dendritic austenite crystallization and the distribution of graphite nodules, as well as refining the primary austenite grains. During the heat treatment of ADI, vanadium carbides, after their dissolution in austenite and dispersion from the metal matrix during isothermal hardening, should strengthen the material. It should be mentioned that according to Goldstein [13], vanadium carbides VC are characterized by a cubic lattice, while V_2C carbide crystallizes in a hexagonal lattice. Thus, VC carbides can be pads for the crystallization of austenite, and V_2C for graphite.

Another form of introducing initiators controlling the crystallization of austenite is iron introduced into a liquid alloy in the inoculation technique, e.g., with a flexible pipe (steel tube) filled with a inoculant. The dissolved steel tube introduces pads with a lattice identical to austenite into the bath, and the introduced modifier controls the crystallization of ball-shaped graphite precipitates [14].

In the presented analogies of the reinforcement of hot-rolled steel and ADI, the strengthening of the structure by dislocations should be omitted. However, in ADI, during the tensile test, the TRIP (Transformation Induced Plasticity) phenomenon occurs, affecting the strengthening of the material [15, 16], generating a martensitic transformation. This is indicated, according to Nofal [15], by an increase in the instantaneous value of the strain hardening exponent during tensile deformation. The TRIP phenomenon also occurs in the micro-areas of the exploited castings surfaces.

2. Scope of research

The research was carried out in industrial conditions. Cast iron was melted in an induction furnace with a crucible capacity of 7 Mg. Alloying elements (Cu and Ni) were introduced into the furnace together with a charge consisting of pig iron (40%), steel scrap (20%) and ADI recycled cast iron scrap (40%). The liquid alloy was taken in portions of 1 Mg, poured into a slender ladle, where a spheroidizing Nodulizer alloy with 6.5% Mg content was introduced using the Thundish Cover method. The alloy with magnesium was poured into a pouring ladle and modified with a barium modifier in the amount of 0,4% of the alloy mass. The temperature was measured, samples were taken for spectral analysis and ATAS thermal analysis.

The following types and weights of additives were added into the liquid alloy in each ladle:

Ladle 1: no additives,
Ladle 2: cast iron shavings 4 kg from casting machining (addition to the Mg casting ladle),
Ladle 3: steel shavings 4 kg (addition to the Mg casting ladle),
Ladle 4: Fe-Nb 2 kg with a grain size of 2-6 mm (including bar modifier),
Ladle 5: Fe-V 2 kg with a grain size of 2-6 mm (including bar modifier),
Ladle 6: pre- inoculation in Ba10 furnace + 2 kg Fe-V,
Ladle 7: pre- inoculation in the Ba10 furnace.

Pre-inoculation was carried out with barium alloy containing approx. 10% of Ba by mass. The liquid alloy prepared in this way was poured into the moulds in the automatic moulding line. Secondary inoculation was carried out on a stream of cast iron poured into the mould with Ultraseed at about 0.15% mass. The metallurgical quality of the liquid cast iron was controlled using the ATAS system (stationary installation), taking a sample of the liquid alloy from each ladle before pouring it into the moulds. Samples were taken for spectral analysis. Thermal analysis was carried out at the end of pouring of each ladle using a mobile 4-position ATAS system. The first sampler was analysed without adding a modifier, the remaining samplers were filled with Ultraseed modifier weighing 0.15% of the sampler capacity. In each commercial casting mould, a test ingot of Y25, according to EN 1564, was poured. A sample was taken from the ingot to assess the microstructure in the poured state, after heat treatment and to determine the mechanical properties. The samples were heat-treated together with commercial castings, and then the samples were prepared for tensile testing. The graphite morphology was assessed using an optical microscope (NIKON 100), the microstructure was examined with electron microscopy and the EDS system for recording spatial distribution maps, Tescan model VEGA3.

3. Test results

Table 1 presents data on the chemical composition and properties of cast iron from individual research experiments. The post-furnace treatment ensured the appropriate level of magnesium in the cast iron. The amount of V and Nb modifiers introduced was kept at the level of micro-additives. The CE carbon equivalent of cast iron was obtained in accordance with the technological recommendation for the produced castings. The introduction of the premodifier resulted in an increase in the CE value (silicon effect). The proportion of trace elements (TE) increased with the addition of a steel shell and a premodifier. The R_m strength values increased compared to cast iron without additives, especially after the introduction of vanadium and pre- inoculation. However, this effect was achieved at the cost of yield strength and elongation.

Table 1.
Characteristics of cast iron subjected to various inoculation treatments

Additives	Time [h:min.]	S [%]	Mg [%]	V [%]	Nb [%]	TE*	CE**	R_{p02} [MPa]	R_m [MPa]	A [%]	HBW
No additives	0:00	0,010	0,044	0,008	0,002	1,563	3,98	579	966	16,0	274
Iron chips 4 kg	0:16	0,008	0,048	0,008	0,002	1,425	4,04	596	1001	16,3	289
Steel chips 4 kg	0:34	0,011	0,046	0,009	0,003	1,016	4,14	600	1022	17,4	289
Fe-Nb 2kg	0:48	0,008	0,042	0,009	0,016	1,344	4,15	608	975	12,1	283
Fe-V 2 kg	1:07	0,008	0,048	0,069	0,015	3,318	4,15	552	1023	12,3	280
Ba10+Fe-V 4 kg	1:21	0,010	0,044	0,102	0,013	1,600	4,20	515	1072	11,9	294
Ba10 5 kg	1:37	0,009	0,054	0,020	0,007	2,392	4,20	479	1079	13,3	285
**Carbon Equivalent CE = C + 0,31 Si											
*Trace Elements TE = 4,4 * %Ti + 2 * %As + 2,3 * %Sn + 5 * %Sb + 290 * %Pb + 370 * %Bi + 1,6 * %Al											

The number of graphite precipitations in cast iron was assessed, both in its poured state and after heat treatment, (Fig. 5). Cast iron kept in a melting furnace for over 1.5 hours, usually loses its ability to graphitize. The introduction of a steel shell and micro-additives stabilized the high ability of the liquid alloy to graphitize in the cast state. In the heat-treated castings, the number of graphite precipitates was smaller. The fewest precipitates were observed in cast iron with the addition of niobium.

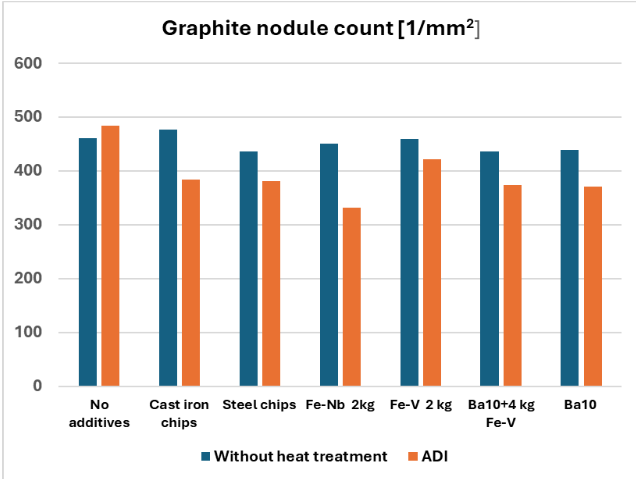


Fig. 5. The influence of Liquid Alloy Additives on the Number of Graphite Nodules in Castings

Investigations of the structure of cast iron in the cast state showed the microstructure of fine pearlite (Fig. 6a) with slightly increased dispersion in the case of cast iron with micro-additives (Fig. 6b, 7). In the pearlite of cast iron with micro-additives, fine particles of a few µm in size are visible, identified as vanadium and niobium compounds (Fig. 6c, 8)

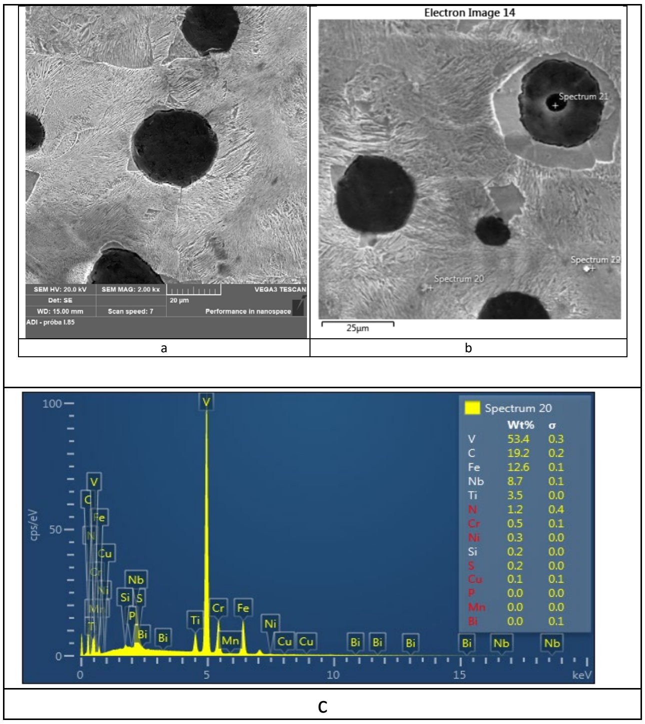


Fig. 6. Microstructure of cast iron in the cast state: a) cast iron without additives, b) cast iron with vanadium and niobium

The identified precipitates are shown in Fig. 7, and their exemplary EDS analysis in Fig. 8

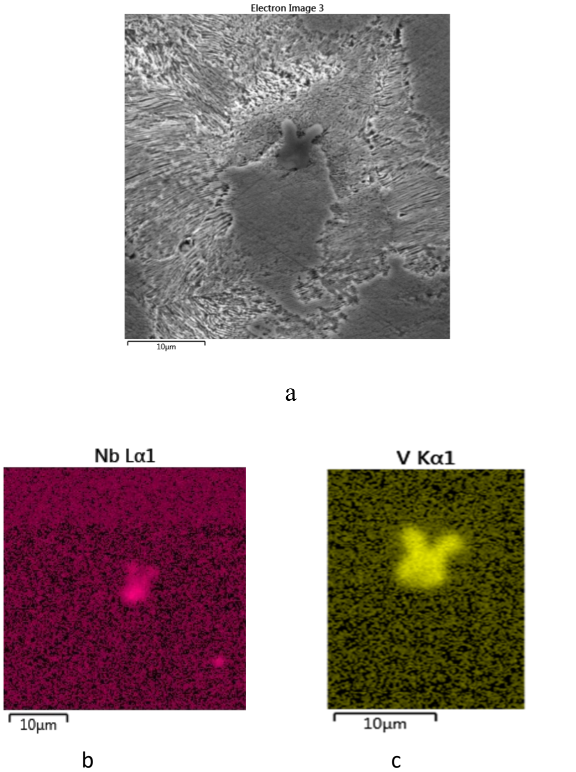


Fig. 7. Microstructure of cast iron in the cast state a) pearlite with precipitations, maps of spatial distribution: b) Nb, c) V

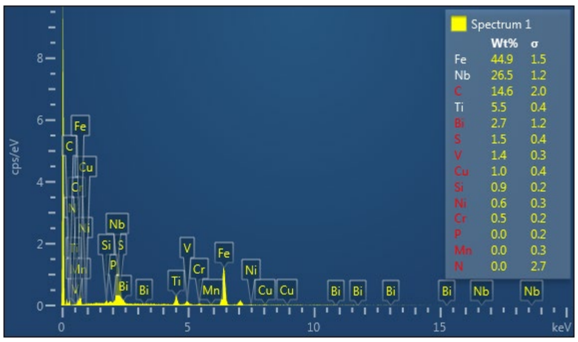


Fig. 8. Identification of the precipitate in cast iron (Fig. 7)

Unlike nitrogen-enriched steel [4-6], cast iron is dominated by carbides.

The microstructure of cast iron after heat treatment is shown in Fig. 9.

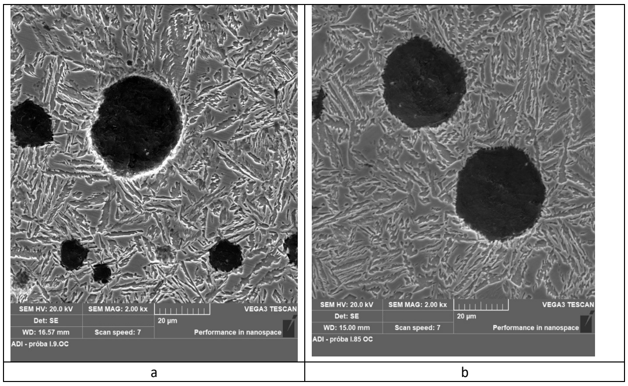


Fig. 9. Microstructure of ADI after heat treatment: without V and Nb (a) and with V and Nb (b)

Ausferrite of cast iron with micro-additives is characterized by greater dispersion and a slightly lower share of block austenite. At comparable magnifications, after heat treatment, such as austenitizing, no additional precipitations were detected in the structure compared to the as-cast state.

Local measurement of the chemical composition performed by the EDS method indicated the presence of vanadium and niobium carbides in the ausferrite matrix in the positions market in Fig. 10. The results of the EDS analysis for these areas are shown in Fig. 11 and Fig 12.

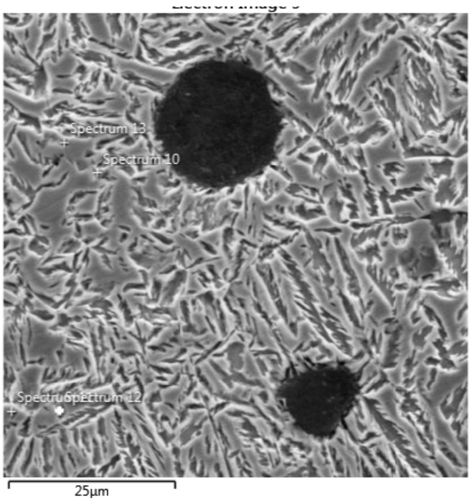


Fig. 10. Microstructure of cast iron with V i Nb; graphite precipitation in ausferrite matrix

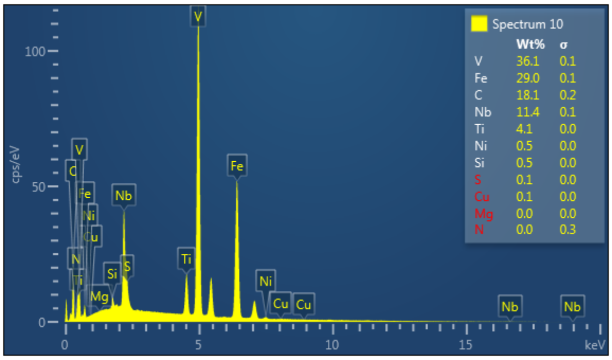


Fig. 11. Identification of precipitates (EDS); V i Nb carbides

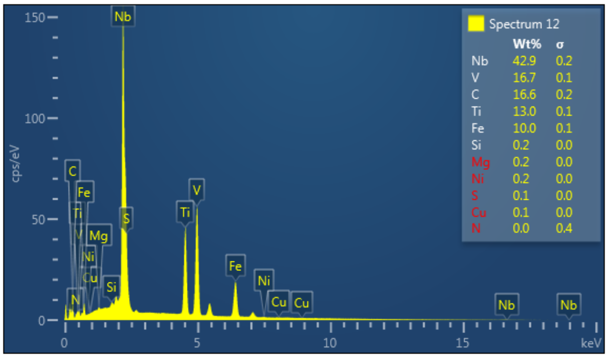
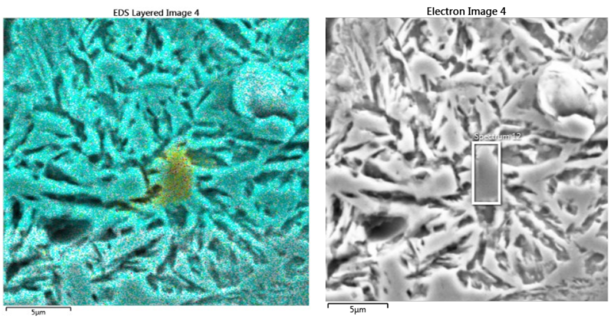


Fig. 12. Identification of precipitates (EDS); V i Nb carbides



a b

Fig. 13. Vanadium precipitates in ausferrite: a) dispersive precipitates, b) preparation of the precipitates for EDS analysis

The EDS analysis of the "cloud" of precipitates is shown in Fig. 14 at a magnification of approximately 26,000. The precipitates were nano-sized clusters, difficult to identify using point EDS analysis. The analysis of the separated area was successfully used (Fig. 13b), and its results are shown in Fig. 14. Vanadium dominates in the precipitates

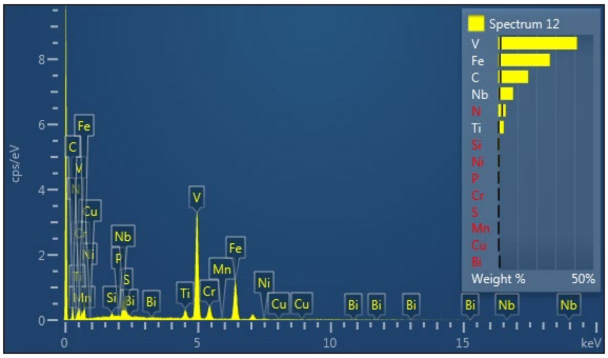
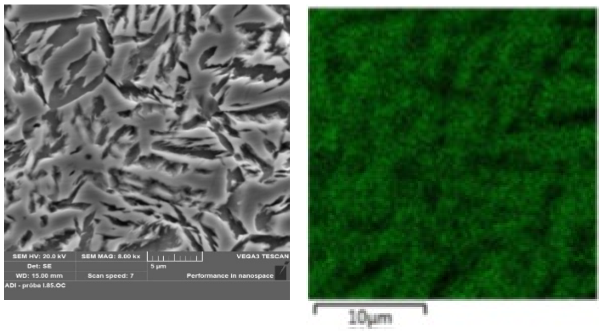


Fig. 14. EDS analysis of the area from Fig. 13a

V carbides are more susceptible to dissolution at austenitizing temperatures in both steel [4,5], cast steel [7], and cast iron [3]. After lowering the temperature, they may be released again at the temperature of final steel rolling, aging of cast steel or ADI. Nano-sized precipitates in ausferritizing cast iron with Nb were also identified by W. Scundlarek et al. [10].

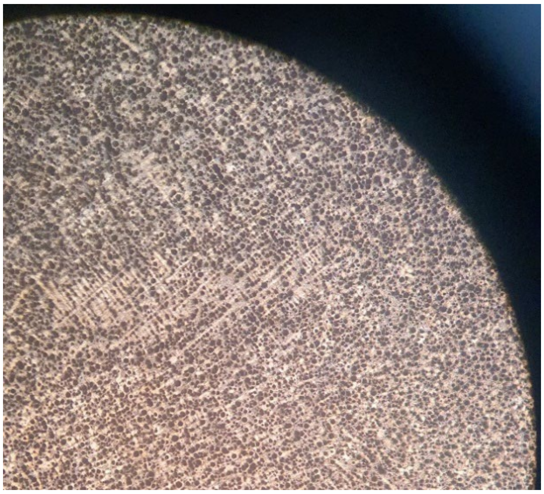
The distribution of silicon in the ausferrite was identified (Fig. 10 a, b), indicating its micro-segregation.



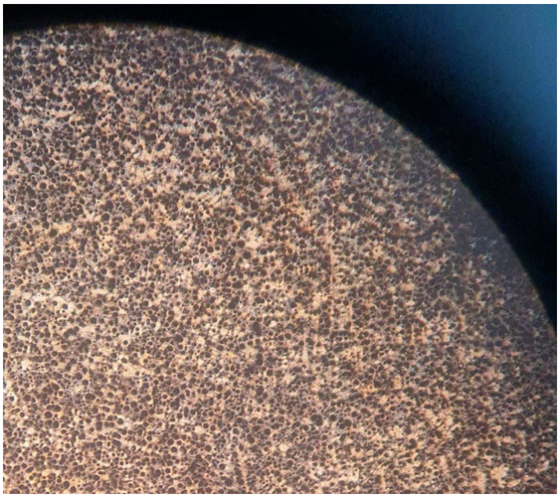
a b

Fig. 15. ADI microstructure: a) ausferrite, b) silicon micro-segregation.

The impact of micro-additives on the morphology of the macrostructure was found, confirming their modifying effect (Fig. 16). A similar effect of micro-additives was found in low-manganese cast steel [7]



a



b

Fig. 16. Macrostructure of ADI: a) without additives, b) with 0.69% V and 0.015% Nb content (etched with Oberhoffer's reagent)

4. Analysis of results

The solidification process of ductile cast iron is the crystallization of nodular graphite and dendritic crystallization of austenite. The amount of graphite precipitation and its nodularity were obtained by appropriate Mg treatment and selection of modifiers. The segregation of alloying elements Ni and Cu in cast iron intended for ausferritic can be mitigated by refining the austenite dendrites [3]. For this purpose, iron alloy chips were used to provide bases for austenite crystallization (crystal lattice compatibility) and microadditions V and Nb as proven additives refining the primary austenite grain and weakening its growth during heat treatment (austenitizing) [4 – 7]. Microadditions V and Nb refined the dendrites in cast iron (Fig. 16), similarly to what was found in the case of low-alloy manganese cast steel [7]. Microadditions of V, Nb and Ti are used in the material engineering of microalloyed steel in precipitation strengthening occurring during their hot plastic working. Dissolution of primary precipitations of carbonitrides of these elements in high-temperature rolling processes and then dispersive precipitation at the temperatures of final rolling guarantee high mechanical properties of these products [5, 6]. The thesis of the occurrence of the precipitation strengthening process in the austenitizing and austempering process was confirmed in the conducted studies. Vanadium, used in particular as a temperature-possible in heat treatment of ADI (Fig. 4) [13], proved to be effective. In addition to the fact that it crushed the dendritic structure, it formed carbides in the cast iron in the cast state with sizes of several µm. It can be concluded that the carbides were also the nuclei of graphite crystallization (Fig. 2) [11], which was confirmed in our own studies (Fig. 6 – 8). In the austenitizing process, the precipitates could dissolve in austenite, to precipitate again in the austempering process in nano-sized sizes (Fig. 10–14). This mechanism was indicated by, among others, Sckudlarek et al. [10] and Nofal [15]. The effect of precipitate strengthening was confirmed in mechanical tests of cast iron without and with microadditives (Table 1), in the case of Rm even by over 10%. Heat treatment of austenitic cast iron also affects the morphology of spheroidal graphite, reducing the number of precipitates (Fig. 5). Analysis of the number of precipitates in individual spheroid size classes indicates that graphite below 15 µm disappeared the most after heat treatment. It can be concluded that at austenitization temperatures the finest graphite was dissolved in austenite. This process was confirmed by J. Furmanek et al. [16].

5. Conclusions

The conducted studies show that:

1. The introduction of modifying additives into the liquid alloy has a positive effect on primary crystallization, weakening the crystallization of dendritic grains and segregation of elements in the cast iron,
2. The addition to the liquid alloy of the steel envelope (chips) has a positive effect,
3. compared to cast iron without additives, on increasing strength and plastic properties,
4. Pre-inoculation treatment in the Ba10 furnace and inoculation of cast iron with vanadium, results in an improvement in tensile strength by 11%, but this strengthening negatively affects elongation (by 25%) and R_{p02} value,
5. The number of graphite bead separations, both in the poured state and after heat treatment, is stable, especially for cast iron held in the furnace for more than 1 hour,
6. Heat treatment of cast iron reduces the number of graphite precipitates compared to the poured state,
7. Vanadium and niobium carbides obtained, identified in the structure in the poured state and comminated after austenitizing and austempering,
8. The change in the morphology of carbide-nitride V and Nb precipitates under heat treatment and the increase in strength indicates the occurrence of precipitate strengthening in cast iron.

It should be noted that the same heat treatment parameters were used for all 7 test experiments. Our own research shows [1, 2] that the kinetics of changes in the mechanical properties of ADI are significantly affected by heat treatment parameters.

In particular, micro-additives of vanadium and niobium can have a significant effect on strength and plastic properties. The nature of such changes has been confirmed in low-alloy steels with micro-additives [4-6]. Thus, there is a need to select parameters, in particular the austenitizing temperature and austempering time of ADI.

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