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The Effect of Heat Treatment on the Microstructure and Hardness of an Austenitic Matrix of High-Manganese Cast Steel with the Addition of Molybdenum

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Abstract

The greatest influence on the wear of tool steel has its microstructure, which depends on the chemical composition and heat treatment. The presence of carbides in the alloy matrix is not always desirable and can have an adverse effect on the wear mechanism of this material, resulting in the formation of stresses and even cracks during operation. Therefore, it is necessary to apply heat treatment, which makes the microstructure homogeneous or allows for the precipitation of secondary carbides strengthening the matrix. The main aim of this study is to examine the effect of molybdenum addition on the structure and microhardness of high-manganese cast steel in the as-cast state and after heat treatment. The as-cast microstructure consists of a high-manganese austenitic matrix with molybdenum carbides and alloy ledeburite distributed at grain boundaries. As a result of solution heat treatment, only the alloy ledeburite is dissolved. The result of aging is not the precipitation of secondary molybdenum carbides but of alloy cementite. Raising the temperature or extending the time of solution heat treatment changes the hardness of austenite to a very small degree only, and the decrease in hardness becomes less significant with the increasing addition of molybdenum. Extending the tempering time has a similar effect, and changes in the hardness decrease are less pronounced.

Keywords: Microstructure, Microhardness, Austenite, Alloy cementite, Solution heat treatment, High-manganese cast steel

1. Introduction

In the mining, quarrying and aggregate processing industries, steel castings are used in conditions of simultaneous abrasive wear and impact stresses. Difficult working conditions are the reason why alloys used for machine and device parts must be characterized by high resistance to dynamic loads, high plasticity and low wear. Currently, the material used for components exposed to wear under the effect of high and dynamic unit pressures is heat-treated, high-manganese cast steel, also known as cast Hadfield steel [1-5].

The functional properties of cast Hadfield steel depend on the parameters of the applied heat treatment as well as on the content of carbon, manganese and carbide-forming additives [3, 4, 6, 7]. This means that there are many standardized grades available on the market, which differ in their chemical composition and production technology, which in turn is determined by the thickness of the casting wall [7]. Currently, research and development activities focus on technological solutions that increase the abrasion resistance of high-manganese cast steel.

In the as-cast condition, Hadfield steel consists of an austenitic matrix, lamellar alloy cementite at grain boundaries, triple phosphorus eutectic of the Fe-$(Fe,Mn)_3C$-$(Fe,Mn)_3P$ type, which appears when the phosphorus content exceeds 0.04%, and non-metallic inclusions in the form of oxides, sulphides or nitrides [1, 8, 9]. The presence and morphology of $(Fe,Mn)_xC_y$ type carbides significantly affect the wear resistance of Hadfield steel castings [10]. The segregation of elements and the formation of alloy cementite precipitates contribute to the matrix impoverishment in alloying elements with the resulting formation of internal stresses [1, 8, 10]. Studies show that the precipitation of carbides at grain boundaries can cause a tenfold reduction in impact strength and a significant reduction in the ductility of cast Hadfield steel products [1, 8, 10, 11]. The decrease in functional properties justifies the need for heat treatment [1].

Traditional heat treatment of cast Hadfield steel consists of solution heat treatment in the temperature range of 1050-1150°C followed by cooling in water [3, 12]. Correlation of appropriately selected heat treatment parameters, i.e. solution heat treatment temperature and cooling rate, allows obtaining a purely austenitic matrix, which, being free from carbide precipitates, is both plastic, i.e. resistant to crack formation, and capable of strong surface hardening under the effect of impacts [4, 11-13]. The mechanism of austenite hardening results from the interaction of slip, the potential of high-alloy austenite to twin in the early stages of deformation and its ability to undergo diffusionless transformation due to the accumulation of dislocations [14, 15]. As a result of stress caused by, for example, a strong impact, the energy absorbed by the steel makes the atomic lattice of the crystal shift with the resulting deformation of the lattice. As a result of repeated slips, dislocations accumulate and pile up, which results in the formation of twin deformations [15]. Twins locally increase the mechanical properties of the surface layer, including tensile strength, hardness and yield strength, while inner layers, including the core, remain plastic [4, 16, 17].

At low dynamic loads, the abrasive wear resistance of cast Hadfield steel is low and comparable to the wear resistance of medium-carbon cast steel. This is due to the lack of proper factors which would enable its surface to be strengthened by cold work and plastic deformation in the working zone [1, 3, 10, 18, 19].

It should be noted that the homogeneous microstructure obtained after heat treatment does not guarantee sufficient abrasion resistance under abrasion conditions. Therefore, technologies are used that allow the precipitation of carbides, but only within the austenite grains and not at grain boundaries. Such solutions operate either without modification of the chemical composition, i.e. by using two-stage heat treatment [2], or with modification of the chemical composition, i.e. by introducing carbide-forming elements that form secondary carbides.

Two-stage heat treatment consists of a long-term stage of isothermal annealing and austenitizing. The material obtained as a result of isothermal annealing is cast steel characterized by an austenitic structure with carbides and products of austenite diffusion transformation, i.e. precipitates of fine-grained pearlite colonies located at grain boundaries. In turn, re-austenitization causes recrystallization of the cast steel, which results in the formation of a fine-grained austenitic matrix with spherical carbide precipitates evenly distributed in this matrix [2, 4]. This structure promotes an increase in abrasion resistance in the range of 6-16% compared to cast Hadfield steel samples subjected to traditional, one-stage heat treatment.

Another method of increasing the abrasion resistance of high-manganese cast steel is to produce a two-phase structure with carbide precipitates by introducing vanadium [20], titanium [1] [13] or niobium [21] into the alloy in the final phase of melting. The addition of each of these elements results in the formation of primary carbides evenly distributed in the austenitic matrix, while the use of heat treatment, which involves solution heat treatment, contributes to solution strengthening of the alloy matrix. The abrasion resistance of such alloys depends on the amount of precipitated primary carbides and their tendency to segregate. Therefore, depending on the amount of introduced carbide-forming elements, different degrees of wear are obtained compared to cast Hadfield steel with a conventional chemical composition, though even a small addition of V, Ti and Nb significantly increases the abrasion resistance.

In addition to vanadium, titanium and niobium, carbide-forming elements such as chromium or molybdenum are added to high-manganese cast steel. The addition of chromium ranges from 0.5 to 2.5% and leads to the precipitation of complex carbides, both at grain boundaries and inside the grains. Increasing chromium content in the alloy increases the amount of alloy cementite [7, 8]. The high thermodynamic stability of cementite $(Fe, Mn, Cr)_3C$ and the relatively large addition of chromium prevent complete dissolution of carbides, which adversely affect the functional properties of high-manganese cast steel with Cr [8]. Obtaining a purely austenitic matrix would involve increasing the temperature and extending the heat treatment time, which would significantly increase production costs and could cause austenite grain growth, and thus reduce both mechanical properties and abrasion resistance [10, 12]. It should be noted, however, that grain growth during heat treatment is insignificant, and factors determining the grain size in castings are primarily of the technological nature related to the preparation of liquid metal and thickness of the casting wall [3].

While the effect of chromium, which is the most commonly used alloying additive, on the structure and properties of high-manganese cast steel has been widely studied, there is little information on the effect of molybdenum when added in significant amounts. In the studies of high-manganese cast steel, the addition of molybdenum is up to 2%, but in foundry practice, due to its high price, this addition is most often limited to tenths of a percent and does not exceed 1% [3, 5, 7, 19]. In [3, 7, 19], with the increase in molybdenum content, a gradual decrease in the size of austenite grains was observed, which was considered to be associated with the precipitation of molybdenum carbides inhibiting the austenite grain growth. In general, the addition of molybdenum has a positive effect on mechanical properties, including hardness, impact strength and abrasion resistance, the latter of which increases after heat treatment.

The current literature review has shown a lack of information on the effect of molybdenum additions of more than 1% on the performance, wear resistance and heat treatment of high-manganese cast steel. Therefore, the aim of this study is to investigate the effect of various heat treatment parameters on the structure of high-manganese cast steel with molybdenum additions of about 2.7%, 3.7% and 5.8 wt.% Mo.

2. Materials and Methods

Test melts were made in a Balzers laboratory vacuum induction furnace with a crucible of about 1 kg capacity. Cast Hadfield steel with a known chemical composition (see Table 1) and Fe-Mo60 were used as a melting charge. After melting the charge, the metal bath was deoxidized with Fe-Si-Ca30 in the amount of 1.0 g/kg, and then Fe-Mo was added in portions. After dissolution of the alloying additive, mixing the melt, heating to a temperature of about 1540°C and temperature equalization, the liquid steel was deoxidized again with Al in the amount of 0.6 g/kg. The metal prepared in this way was poured into ceramic moulds heated to about 200°C. In this way, three "Y" type test ingots with a wall thickness of 30 mm and a weight of about 1 kg were obtained. Depending on the Fe-Mo60 addition, each of the ingots contained a different molybdenum content. The chemical composition of the obtained ingots, which is presented in Table 1, was examined with a SPECTROMAXx spark spectrometer. Compared to the standard chemical composition of cast Hadfield steel (GX120Mn13), the obtained castings were characterized by carbon content increased to about 1.4 wt.%, manganese content of about 14 wt.% and silicon content of 1.3-1.6 wt.%, which resulted from the deoxidation of liquid steel with Fe-Si-Ca30. The chromium content of about 0.3 wt.%, a low content of other elements, such as Ni, Al and Ti, and the content of impurities, i.e. sulphur and phosphorus, at the level of hundredths of a percent had no significant effect on changes in the functional properties of the ingots. In the article, the proposed molybdenum contents were selected based on the average molybdenum addition to tool steels, which ranges from 1% to 6% Mo, and allows for the precipitation strengthening of the austenitic matrix during heat treatment. Three different molybdenum contents were obtained in the tested samples, i.e. 2.7%, 3.7% and 5.8 wt.%, respectively.

Samples for testing were cut out from the test ingots. Some of the samples were left in the as-cast state, while other samples were subjected to heat treatment, which involved solution heat treatment at a temperature of 1100°C, 1150°C and 1200°C for 90 minutes or 180 minutes with cooling in a 10% aqueous solution of HO polyhartenol, and then aging of selected samples at 600°C for 120 minutes, 120+60 minutes, and 120+60+60 minutes. The aim of the selected type of heat treatment was to determine the effect of solution heat treatment temperature on the rate of dissolution of molybdenum carbides and the effect of aging time on the precipitation strengthening of an austenitic matrix.

After each heat treatment operation, the microhardness of the austenitic matrix was measured by the Vickers method using a Prazision HV1000B hardness tester under a load of 200 g. The average hardness of the austenitic matrix was calculated from at least eight measurements. Figure 4 and Figure 7 show the heat treatment parameters and the average hardness of the matrix along with the deviation error.

From the tested samples in the as-cast state and after heat treatment, metallographic sections were prepared, which were next etched with a 5% solution of nitric acid in ethyl alcohol. The microstructure of the tested samples was examined using a Kern Optics light microscope at a magnification of 200x and 500x. This microscope is equipped with a camera and a system for automatic, digital image recording.

The morphology of carbides, alloy cementite and alloy matrix was examined with a JEOL 5500 LV scanning electron microscope, which is equipped with a microanalyzer and an EDS detector to perform an analysis of the chemical composition of the occurring phases.

Table 1.
Chemical composition of charge, reference cast Hadfield steel and test ingots.

Designation	Content of elements [wt.%]									
	C	Mn	Si	P	S	Cr	Ni	Al	Ti	Mo
Charge for melting	1.67	14.4	1.45	0.05	0.02	0.34	0.01	0.02	0.03	0.01
Reference GX120Mn13	1.2	13	0,8	0.03	0.02	-	-	-	-	-
MnMo2.7	1.48	13.83	1.62	0.05	0.01	0.31	0.02	0.03	0.03	2.7
MnMo3.7	1.46	13.40	1.59	0.05	0.02	0.30	0.02	0.03	0.02	3.7
MnMo5.8	1.38	12.92	1.26	0.04	0.02	0.27	0.02	0.02	0.03	5.8

3. Test results and discussion

The article presents the results of research on the obtained microstructure of high-manganese cast steel with additions of molybdenum in the amount of 2.7%, 3.7% and 5.8 wt.% Mo in the as-cast state and after heat treatment, as well as the results of measurements of the microhardness of an austenitic matrix of the tested alloys.

3.1. Microstructure in the as-cast state

Based on observations carried out by light microscopy (Fig. 1), scanning microscopy (Fig. 2) and chemical analysis of the visible precipitates and matrix (Table 2), it was found that the microstructure of the tested cast steel in the as-cast state consists of a high-manganese austenitic matrix (Table 2, point 4, Fig. b-c)), in which, after the solidification process, molybdenum carbides of various stoichiometry ($((Mo,Fe,Mn)_xC_y)$, (Table 2, point 1, Fig. b-c)) are the first ones to precipitate at grain boundaries in the form of irregular plate-shaped and oval particles. At a later stage of cooling, ledeburite plates nucleate, precipitate and grow on molybdenum carbides as a mixture of austenite and alloy cementite $(Fe, Mn, Mo)_xC_y$ - in the form of acicular and plate-like, branched precipitates (Table 2, points 2 and 3, Fig b-c)). It was observed that the amount of precipitated alloy cementite decreases with the increasing addition of molybdenum, in favour of the molybdenum carbides, the content of which increases. The largest amount of cementite, which is responsible for binding the largest amount of carbon from the matrix, was observed for the Mo addition of 2.7 wt.%, and the smallest for the content of 5.8 wt.%. Moreover, it was shown that the addition of molybdenum results in the refinement of primary austenite grains, the degree of this refinement being directly proportional to the amount of molybdenum introduced to the alloy. The refinement of the primary structure has been confirmed by the results of research presented in [3, 19].

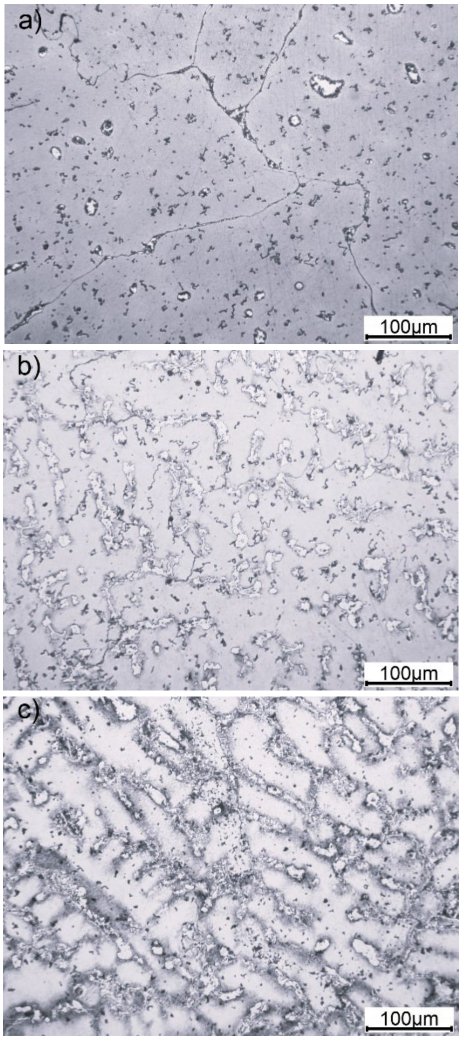


Fig. 1. Examples of the microstructure of high-manganese cast steel with the addition of: a) 2.7%, b) 3.7%, c) 5.8 wt.% Mo, as-cast state, etched with nital, austenitic high-manganese matrix with precipitates of carbides $(Mo,Fe,Mn)_xC_y$ and alloy cementite $(Fe, Mn, Mo)_xC_y$

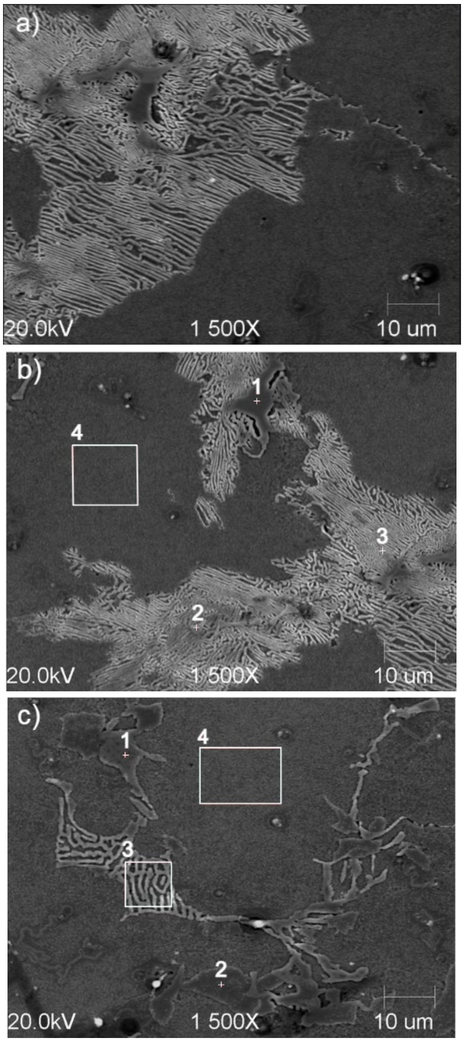


Fig. 2. Examples of the SEM microstructure of samples of the tested alloys containing respectively: a) 2.7%, b) 3.7%, c) 5.8 wt.% Mo, etched with nital, austenitic high-manganese matrix with precipitates of carbides $(Mo,Fe,Mn)_xC_y$ and alloy cementite $(Fe, Mn, Mo)_xC_y$

Table 2.
Chemical composition of characteristic points 1,2,3,4 in Fig. 2.

	Location of analysis	[at%]					
		C	Cr	Mn	Mo	Si	Fe
Fig. b)	point 1	62.6	0.6	5.6	25.2	-	Balance
	point 2	26.8	0.5	17.2	4.6	-	Balance
	point 3	31.5	0.2	11.9	1.1	-	Balance
	point 4	16.6	0.1	6.8	0.8	2.4	Balance
Fig. c)	point 1	63.7	0.7	4.8	25.3	-	Balance
	point 2	32.9	0.3	9.2	0.8	-	Balance
	point 3	43.8	0.4	7.8	8.0	-	Balance
	point 4	24.5	0.3	7.7	0.7	-	Balance

The observed tendency towards precipitation and formation of carbide clusters due to the increased content of the carbide-forming element, which is molybdenum, has been confirmed by the results of earlier studies described in [1, 8, 13, 19, 20]. The precipitation of a large amount of lamellar carbides favours the formation of thermal and structural stresses, not only during cooling of castings, but also during heat treatment, which can cause brittle fracture of cast components. Therefore, it is important to control the rate of temperature changes during individual technological processes. Castings with this microstructure cannot be subjected to dynamic and impact loads during operation, because they have reduced plasticity which, combined with variable stresses, can cause sudden and catastrophic destruction of the structural component. For the above-mentioned reasons, it is recommended to apply heat treatment, which consists of solution heat treatment carried out in such a way as to obtain a purely austenitic matrix, free from carbide precipitates at grain boundaries, which gives the highest resistance to cracking and ensures the highest plasticity of the matrix.

3.2. Microstructure after solution heat treatment

The conducted heat treatment, which involved the solution heat treatment under the following heating temperature/time conditions: 1100°C/90 min., 1150°C/90 min., 1150°C/180 min. and 1200°C/90 min., did not lead to the dissolution of molybdenum carbides, and thus could not guarantee the achievement of a purely austenitic structure (Fig. 3). In a similar way, increasing the treatment temperature or extending the treatment time has also proved to be ineffective in this respect. Based on observations conducted by light microscopy (Fig. 3), it can be concluded that only plate-like precipitates of alloy cementite – (Fe, Mn, Mo)$_x$C$_y$ – were dissolved. Molybdenum carbides (Mo, Fe, Mn)$_x$C$_y$, which appeared at grain boundaries and precipitated as the first constituent in the solidification process, were not completely dissolved. This indicates their high thermodynamic stability, which is the higher, the higher is the content of molybdenum in the alloy. This is confirmed and illustrated by the obtained results of measurements of the microhardness of an austenitic matrix (Fig. 4). Increasing the temperature or extending the time of the solution heat treatment has but only a very insignificant effect on changes in the hardness of austenite, and a decreasing trend was observed in this respect; the hardness of austenite decreased from about 480 HV0.2 in the as-cast state to about 400 HV0.2 after the solution heat treatment carried out at a temperature of 1200°C for 90 min, which may indicate a low rate of the dissolution of molybdenum carbides. It was found that the decrease in hardness was the lower, the higher was the content of molybdenum in the tested alloy.

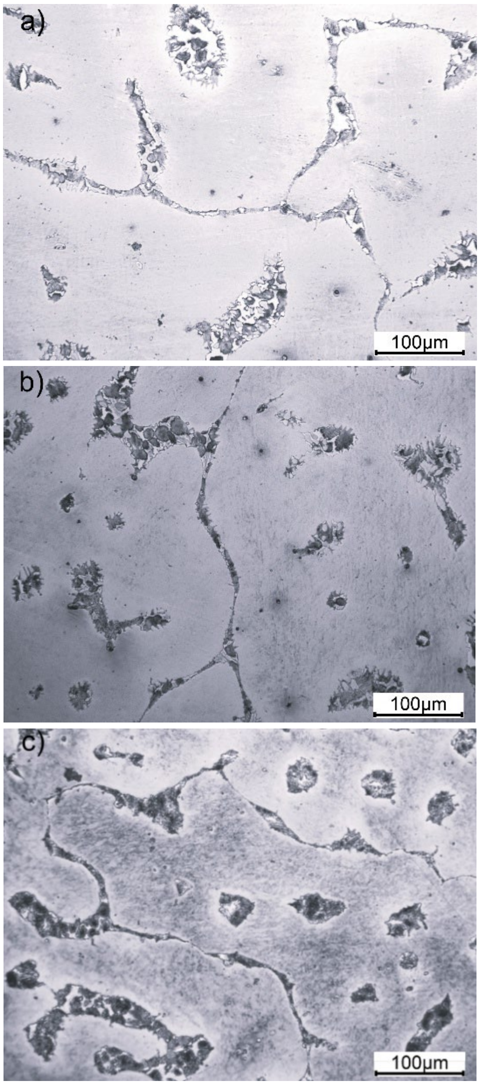
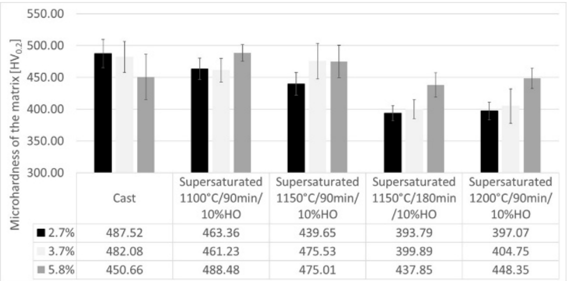


Fig. 3. Examples of the microstructure of samples after solution heat treatment at 1200°C/90 min with well visible, undissolved molybdenum carbides: a) 2.7 %, b) 3.7%, c) 5.8 wt.% Mo, etched with nital, 200x, austenitic high-manganese matrix with undissolved precipitates of carbides (Mo,Fe,Mn)$_x$C$_y$

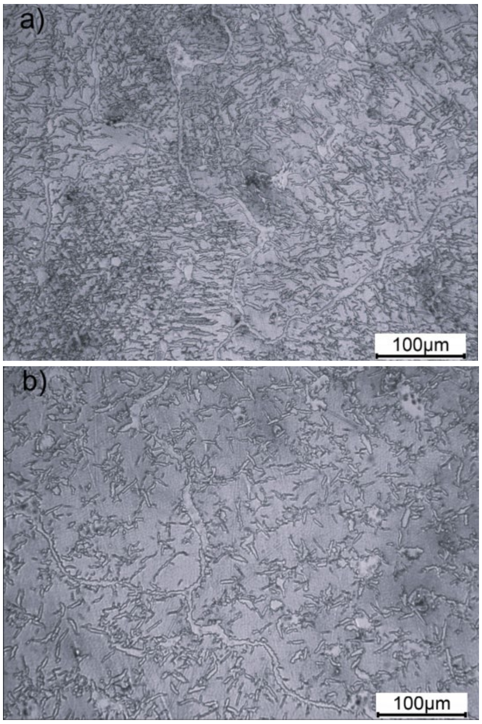


	Cast	Supersaturated 1100°C/90min/ 10%HO	Supersaturated 1150°C/90min/ 10%HO	Supersaturated 1150°C/180min /10%HO	Supersaturated 1200°C/90min/ 10%HO
■ 2.7%	487.52	463.36	439.65	393.79	397.07
□ 3.7%	482.08	461.23	475.53	399.89	404.75
▨ 5.8%	450.66	488.48	475.01	437.85	448.35

Fig. 4. The effect of solution heat treatment parameters on the average microhardness of an austenitic matrix of the tested alloys

3.3. Microstructure after ageing

After solution heat treatment, the samples were aged successively for 120 min, 120+60 min and 120+60+60 min at a temperature of 600°C, and then cooled in the airto obtain the effect of precipitation strengthening of the alloy matrix due to the presence of dispersive molybdenum carbides. Observations carried out by both light microscopy (Fig. 5) and SEM (Fig. 6), as well as the chemical phase analysis (Table 3) showed the presence of undissolved molybdenum carbides of various stoichiometry ($(\mathrm{Mo,Fe,Mn})_x\mathrm{C}_y$) (point 1, Table 3). Precipitates of secondary molybdenum carbides were not observed, but precipitates of alloy cementite $(\mathrm{Fe,Mn,Mo})_x\mathrm{C}_y$, of various stoichiometry (points 3, 4, 7, Table 3) appeared in the form of rods and sticks in the entire volume of the matrix. It was found that with the increasing molybdenum content in the alloy, the amount of the precipitated cementite decreased, and at the content of 5.8 wt.%, the cementite did not precipitate at all (Fig. 5c). The only advantage of the applied heat treatment was changing the morphology of cementite. It precipitated in the form of single sticks and rods, evenly distributed throughout the whole volume of the austenitic matrix, and not in the form of plates which clearly dominated in the as-cast condition (Fig. 2).



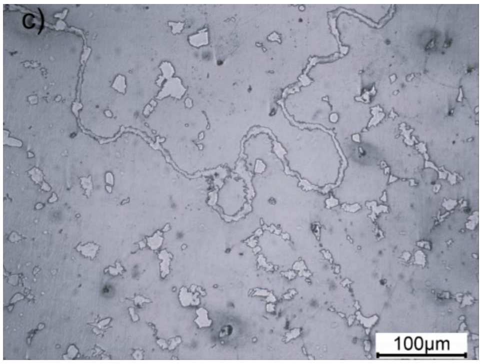


Fig. 5. Examples of the microstructure of samples aged at 600°C for 120+60+60 min; visible is the precipitated cementite; a) 2.7%, b) 3.7%/180 min, c) 5.8 wt.% Mo, etched with nital, 200x, austenitic high-manganese matrix with undissolved precipitates of carbides $(\mathrm{Mo,Fe,Mn})_x\mathrm{C}_y$ and secondary precipitates of alloy cementite $(\mathrm{Fe,Mn,Mo})_x\mathrm{C}_y$ a)-b)

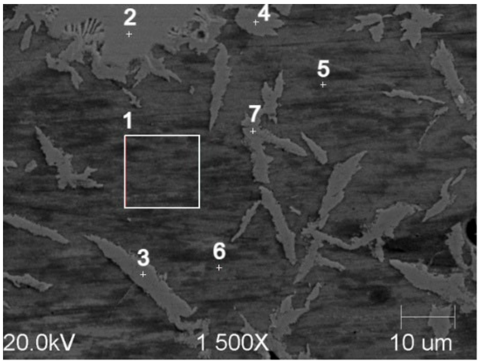
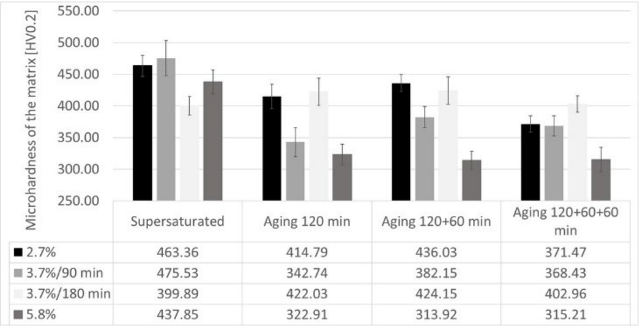


Fig. 6. Example of the SEM microstructure of sample containing 2.7 wt.% Mo, etched with nital, austenitic high-manganese matrix with undissolved precipitates of carbides $(\mathrm{Mo,Fe,Mn})_x\mathrm{C}_y$ and secondary precipitates of alloy cementite $(\mathrm{Fe,Mn,Mo})_x\mathrm{C}_y$

Table 3.
Chemical composition of characteristic points 1-7 in Fig. 6.

Location of analysis	[at%]					
	C	Cr	Mn	Mo	Si	Fe
point 1	17.6	0.2	7.9	0.9	2.2	Balance
point 2	0.1	1.0	19.8	8.5	2.6	Balance
point 3	34.1	0.2	6.1	0.6	1.9	Balance
point 4	0.1	0.2	10.4	1.4	4.9	Balance
point 5	16.9	0.3	8.9	1.0	2.9	Balance
point 6	11.7	0.4	8.9	1.1	2.6	Balance
point 7	6.6	0.2	9.0	0.5	3.3	Balance

During the first 120 minutes of ageing, a strong decrease in the hardness of the austenitic matrix was observed in the tested samples (Fig. 7). Extending the tempering time by additional hours had a similar effect but with less prominent differences in the hardness decrease. The precipitation of carbon and manganese in the form of alloy cementite from austenite reduces the hardness of the matrix. The lowest hardness of slightly over 300HV0.2.was measured in the alloy with the highest molybdenum content.



	Supersaturated	Aging 120 min	Aging 120+60 min	Aging 120+60+60 min
■ 2.7%	463.36	414.79	436.03	371.47
■ 3.7%/90 min	475.53	342.74	382.15	368.43
□ 3.7%/180 min	399.89	422.03	424.15	402.96
■ 5.8%	437.85	322.91	313.92	315.21

Fig. 7. The effect of aging time on the microhardness of alloy matrix

The described phenomena of continuous decrease in the hardness of the austenitic matrix during subsequent heat treatment operations are probably caused by hindered diffusion of alloying elements, due to the significant amount of dissolved molybdenum and a relatively large amount of silicon in the matrix, i.e. about 1% and 2-3 wt.%, respectively. The phenomenon of hindered diffusion in the presence of alloying elements dissolved in the matrix is commonly known as the alloy resistance to tempering heat (high temperature).

The proposed heat treatment did not meet the assumed expectations - molybdenum carbides were not dissolved in the austenitic matrix during solution heat treatment, effectively hindering the process of precipitation strengthening of the alloy matrix during ageing. Molybdenum concentration in the matrix was not observed to change in any way, even with the molybdenum content increased in the alloy and changed parameters of heat treatment. Extending the heat treatment time and increasing the temperature caused changes in the morphology of molybdenum carbides so insignificant that the process was considered to be uneconomical.

4. Conclusions

Based on the obtained test results, including chemical analysis of samples, microstructure examinations, microhardness measurements, and changes in the chemical composition of phases occurring in the tested samples after different heat treatment variants, it can be stated that:

1. the microstructure in the as-cast state consists of a high-manganese austenitic matrix, molybdenum carbides, and alloy cementite,
2. during heat treatment only ledeburite dissolves,
3. solution heat treatment causes a very insignificant decrease in austenite hardness,
4. during aging, alloy cementite precipitates in the matrix of the alloy,
5. ageing brings further decrease in austenite hardness,
6. the applied heat treatment does not allow obtaining a purely austenitic matrix,
7. the proposed heat treatment prevents precipitation strengthening of the austenitic matrix during ageing,
8. because of the obtained microstructure, high-manganese cast steel with Mo addition may have only limited applications, the high price of molybdenum and complicated heat treatment make the production process of high-manganese cast steel with Mo addition uneconomical.

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