



Effect of the heat treatment on the structure and properties of GX12CrMoVNbN9-1 cast steel

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ABSTRACT

Purpose: The paper presents the influence of heat treatment parameters (austenitization and tempering temperature) on the microstructure and mechanical properties of high - chromium martensitic GX12CrMoVNbN9-1 (GP91) cast steel. Moreover, the influence of stress relief annealing at the temperatures of 730 and 750°C on microstructure and properties has been investigated.

Design/methodology/approach: Microstructure of the cast steel was characterized using optical metallography and transmission electron microscopy. Identification of precipitates was made by means of thin foils and extraction carbon replicas. The size of precipitations was determined by Image Pro Plus software. Moreover, the mechanical properties (static tension, hardness and impact energy) have been tested.

Findings: What has been determined is the influence of heat treatment parameters on microstructure and mechanical properties of GP91 cast steel. Heat treatment (hardening and high-temperature tempering) of GP91 cast steel allowed to obtain a fine-grained microstructure of high-tempered martensite with numerous precipitates whose properties met the standard requirements, regardless of the heat treatment parameters.

Research limitations/implications: It is necessary to continue the research to determine description of the microstructure after different heat treatment parameters.

Practical implications: Optimization of the heat treatment parameters from the aspect of using the investigated cast steel for long-term operation in power units designed for working at the so-called supercritical parameters.

Originality/value: The relationship between the heat treatment parameters (austenitization and tempering temperatures) and mechanical properties of high - chromium martensitic GX12CrMoVNbN9-1 (GP91) cast steel was specified. Moreover, the influence of the stress relief annealing parameters on microstructure and properties has been shown.

Keywords: Metallic alloys; Mechanical properties; High – chromium martensitic cast steel; Microstructure; Precipitates

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MATERIALS

1. Introduction

The world-wide legal norms in the field of environmental protection, noticeable especially in Europe, and the related economical factors impose reducing the energy generating costs and limiting the emission of pollutants into the air. The above aims are realized through building new power units and modernizing the existing ones. The most effective way of meeting those pro-environmental aims is raising the thermal efficiency of power units. Obtaining higher thermal efficiency (currently 42-45%, expected level up to 50%) and limiting the emission of gases to the atmosphere, mainly CO₂ and NO_x, requires elevating the temperature and pressure of superheated steam in many elements of boilers and turbines. The temperature and pressure of superheated steam in the new-built power units has risen to the level above 580°C and ≥25MPa, respectively (supercritical and ultra-supercritical parameters of work). For the fulfillment of such high requirements it turned out necessary to develop and introduce high-temperature creep resisting steels, with their mechanical properties higher than the “old” steels used so far, into the widely understood power industry. Many years of extensive research has resulted in developing and implementing in the power industry not only high-chromium martensitic steels of 9-12%Cr, such as: P91, P92, E911, or P122(A), as well as low-alloy bainitic steels: T23 and T24. High properties of the new-found steels were obtained thanks to the optimization of their chemical composition, heat treatment and microstructure [1-5].

Applying super- or ultra-supercritical parameters of steam entailed the development of new grades of steels or superalloys, as well as new grades of cast steels. Steel casts which have been used in the power industry so far (turbine body and cylinders, T-pipes, valve chambers, etc.) made of low-alloy Cr-Mo cast steel or Cr-Mo-V cast steel or non-alloy (carbon) cast steel do not meet those requirements [6-8].

The key role in highly-loaded turbine components, such as: valve bodies, valve chamber, outer and inner casings, etc. is ascribed to steel castings. The usage of up to 600°C requires the improved steel casts of 9-12%Cr. What can be applied here is a cast steel with highly-tempered martensitic microstructure due to its mechanical properties along with higher resistance to thermal shock.

High requirements put to the new-found grades of high-chromium cast steels were as follows:

- the 100 000 hours creep strength of 100 MPa at 600°C;
- good castability and weldability;
- through - hardening capability up to the wall thickness of about 500 mm;
- properties, such as: fracture toughness, low-cycle fatigue strength and long-term toughness corresponding at least to those of the low-alloys, ferritic cast steels currently used up to 565°C.

The above requirements were fulfilled by the new grades of cast steels with their chemical composition similar to that of high-chromium cast steels. As a result of intense research, as a part of EPRI and COST projects, the new high-chromium martensitic cast steels have been worked out and introduced for usage in the power industry - GX12CrMoVNbN9-1 (GP91) and GX12CrMoWVNb10-1-1 (GX12). The above-mentioned cast

steels were developed on the basis of chemical composition of high-chromium martensitic steels: P91 and E911, respectively [9-11]. Relatively short literature information concerning new martensitic cast steels designed for service at supercritical parameters has encouraged the Author to take up the matter of such materials. The aim of performed research was to determine the influence of multi-stage heat treatment on the microstructure and properties of martensitic GX12CrMoVNbN9-1(GP91) cast steel.

2. Material and methodology of research

High-chromium GP91 cast steel came into being as a result of a EPRI research project on the basis of chemical composition of P91 steel. The required chemical composition of GP91 cast steel is given in Table 1.

Table 1.
Chemical composition of P91 steel and GP91 cast steel, %wt [12]

	C	Si	Cr	Mo	V	Nb	N
GP91	0.10	0.20	8.0	0.85	0.18	0.06	0.030
	-	-	-	-	-	-	-
	0.14	0.50	9.5	1.05	0.25	0.10	0.070

Similarly as in the case of other grades of martensitic steels and cast steels, the basic element in the discussed cast steel is chromium. The content of this element on the level of 8.0-9.5% provides proper heat resistance and high-temperature creep resistance in the examined cast steel to the steam temperature of 600-610°C. The addition of molybdenum of around 1% is to strengthen the matrix in solution, it elevates the temperature of recrystallization and counteracts the temper brittleness. Microadditions of vanadium and niobium in combination with nitride or/and carbon form the precipitates of MX type (M=V, Nb; X=C, N) which are highly stable thermodynamically. The MX precipitates hinder the movement of dislocations by anchoring them and thus provide high creep resistance [2,3,5,10].

Developing the microstructure and mechanical properties of the investigated cast steel takes place through hardening and high-temperature tempering. The scheme of a typical heat treatment of steel casts of many tons is presented in Fig. 1.

High content of chromium along with the additions and microadditions of molybdenum, vanadium and niobium is the reason why GP91 cast steel is characterized by a very high stability of supercooled austenite. This allows to obtain the martensitic structure by the air-cooling throughout the cross-section up to the thickness of ca. 80 mm, whilst elements of greater wall thickness require faster cooling, e.g. with oil or hardening polymers. The optimum range of austenitizing temperatures is adjusted so as the M₂₃C₆ carbides and MX nitrides (carbonitrides) could get dissolved in a solid solution. Part of the carbides, mostly NbC rich in niobium, should remain undissolved in austenite, which provides fine grain of austenite and has a favorable influence on plastic properties of the heat-toughened cast steel [3,9,11].

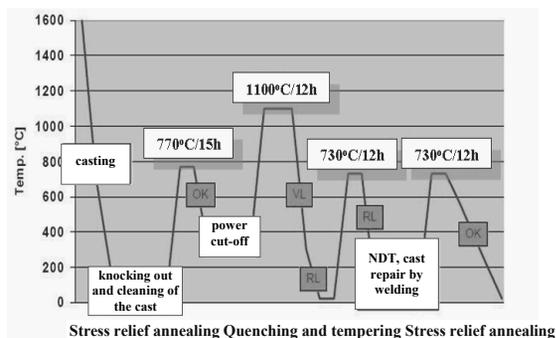


Fig. 1. Heat treatment scheme for high-chromium cast steel, where: OK-cooling with furnace, VL-cooling in compressed air, RL-cooling in calm air [10]

GP91 cast steel after heat-toughening is characterized by the microstructure of high-tempered martensite which should provide minimum mechanical properties included in Table 2.

Applying GP91 cast steel instead of the previously used low alloy Cr-Mo or Cr-Mo-V cast steel enables elevating the steam temperature by 30-50°C.

Table 2.
Required mechanical properties of GP91 cast steel [12]

	$R_{p0.2}$ MPa	TS MPa	El. %	KV J	Creep resistance 100000/600°C, MPa
GP91	min 450	600 - 750	min 15	min 30	86

The material under investigation was a cast steel of the following chemical composition: 0.12C, 0.49Mn, 0.31Si, 0.014P, 0.004S, 8.22Cr, 0.90Mo, 0.12V, 0.07Nb, 0.04N. The chemical composition of the examined alloy corresponded to that of GX12CrMoVNbN9-1 (GP91) cast steel (see Table 1).

Heat treatment of GP91 cast steel included twelve-hour austenitizing of samples at the temperatures of: 1010, 1040, 1070 and 1100°C with the subsequent cooling in oil. The twelve-hour tempering of the samples was performed at three temperatures: 730, 760 and 780°C. After tempering the samples were cooled in calm air. Additionally, for the heat treatment simulation (stress relief annealing), the samples after heat treatment of: 1040°C/12h/oil + 760°C/12h/air, were being held at the temperatures of 730 and 750°C for eight hours and next cooled to the room temperature at the rate ~25 K/h. Microstructural research was performed by means of JOEL JEM-3010 high-resolution transmission electron microscope using thin foils. Identification of precipitates was made by means of thin foils and extraction carbon replicas by electron diffraction. The tests were carried out on samples in the as-cast state and after the heat treatment at the assumed parameters of temperature and time. Mechanical properties were examined according to the currently obeyed

standards. The static tension test was made by means of the MTS-810 testing machine on test pieces with their initial gauge diameter of $d_0 = 8$ mm. Measurement of hardness was taken using the Vickers method with the load of 30 kG (294.2 N), by means of the Future-Tech FV-700 testing machine. Tests of impact energy were carried out on non-standard test pieces of the Charpy V type. In the case of the static tensile test and the impact energy measurement, the presented results are the mean value of three tests, while the hardness value is the mean of five measurements.

3. Microstructure and properties of GP91 cast steel in the as-cast state

The microstructure of GP91 cast steel in the as-cast state was a coarse grain martensitic microstructure with numerous precipitations of carbides (Fig. 2). The cast steel's microstructure in the as-cast state was characterized by the lath structure of martensite with large dislocation density and polygonal substructure (Fig. 2b). The width of martensite laths amounted to ca. 0.41-0.57 μm . In the microstructure on lath boundaries, subgrain boundaries and inside the laths there were numerous precipitates of diverse morphology observed (Figs. 2b, 3).

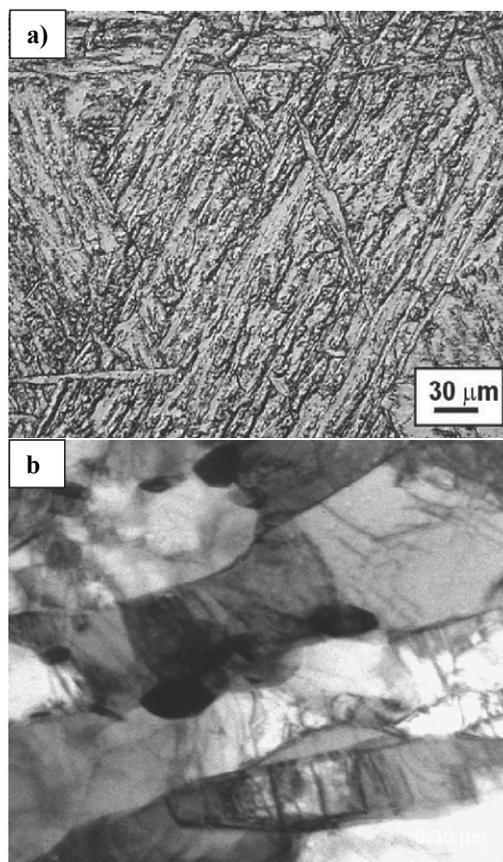


Fig. 2. Microstructure of GP91 cast steel in the as-cast state: a) OM, b) TEM, thin foil

Performed identifications of precipitates in GP91 cast steel by means of electron diffraction revealed the presence of:

- large $M_{23}C_6$ carbides with their mean diameter of about 65-137 nm precipitated mostly on former austenite grain boundaries and martensite laths boundaries. Single precipitates of this type were also observed inside the grains (Figs. 2b, 3);
- fine-dispersion spheroid NbC carbides precipitated on dislocations inside martensite laths as well as on subgrain boundaries (Fig. 2b);
- lamellar M_3C carbides about the size of 40-100 nm precipitated in the Widmannstätten pattern (Fig. 3).

Figure 3 illustrates the morphology of carbides in GP91 cast steel in the as-cast state. Figure 4 shows the characteristic X-ray spectra from the precipitates marked in Fig. 3.

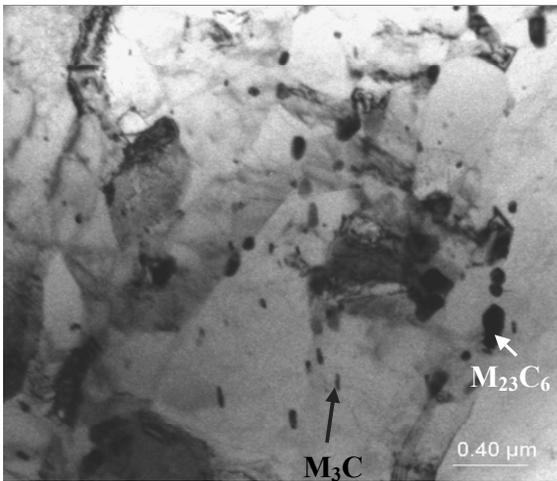


Fig. 3. Morphology of carbides in GP91 cast steel in the as-cast state, TEM, thin foil

High temperature of the beginning phase of martensite transformation M_s , determined for the investigated cast steel on the level of 386°C , makes the carbon diffusion possible. This can lead to the effect of self-tempering of martensite as a result of precipitation of M_3C carbides in the microstructure (Fig. 3). A similar process of martensite self-tempering was also observed in high-chromium martensitic steels, for instance: P92 [3,13]. Numerical simulation of solidification of GP91 cast steel consistent with the Scheil-Gulliver model and performed with the usage of ThermoClac program has revealed that the carbides of niobium NbC (carbonitrides) are precipitated in the final phase of coagulation. NbC carbides, being precipitated thus, are an inhibiting factor for austenite grain growth during heat treatment, which affects further properties of the alloy significantly [4,14].

The scheme of morphology of the precipitates in GP91 cast steel in the as-cast condition is depicted in Fig. 5.

Mechanical properties of GP91 cast steel in the as-cast state are presented in Table 3. The examined cast steel properties in the as-cast state fulfilled the standard requirements. The strength

properties determined for GP91 cast steel in the as-cast condition, such as tensile strength and yield strength were higher than the required minimum by the respective 7 and 2%. While the plastic properties of the investigated cast steel, i.e. impact strength and elongation were respectively: more than three times as high as the minimum and ca. 33% higher than the minimum. Hardness of the examined cast steel in the as-cast state amounted to 232HV30, whilst percentage reduction of area - 58% (Table 3).

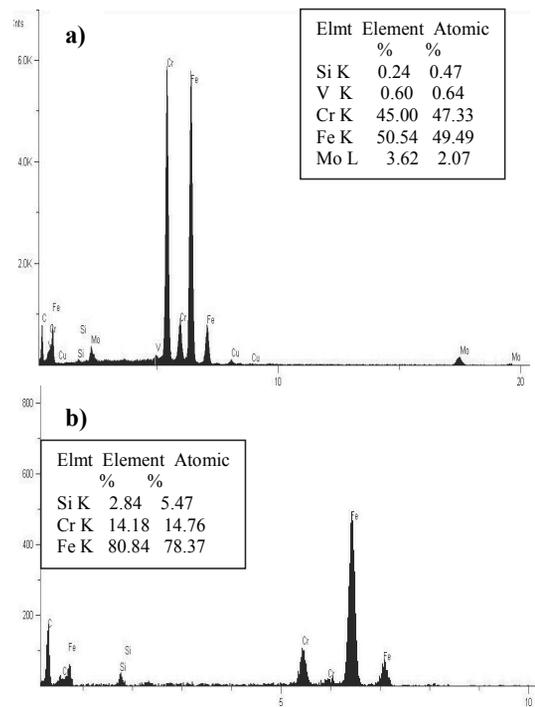


Fig. 4. Examples of characteristic X-ray spectra and concentration of alloy elements: a) $M_{23}C_6$ carbide; b) M_3C carbide (illustrated in Fig. 2)

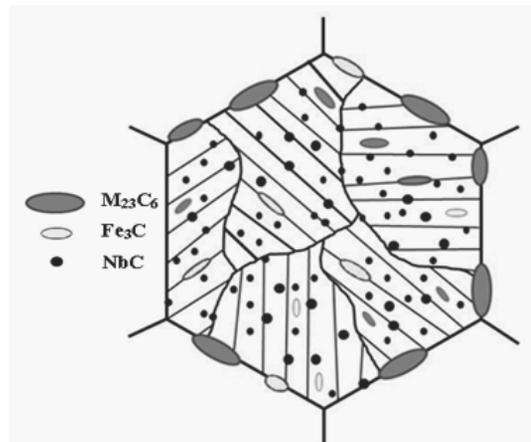


Fig. 5. Schematic illustrations of precipitation morphology in the as-cast condition of the GP91 cast steel

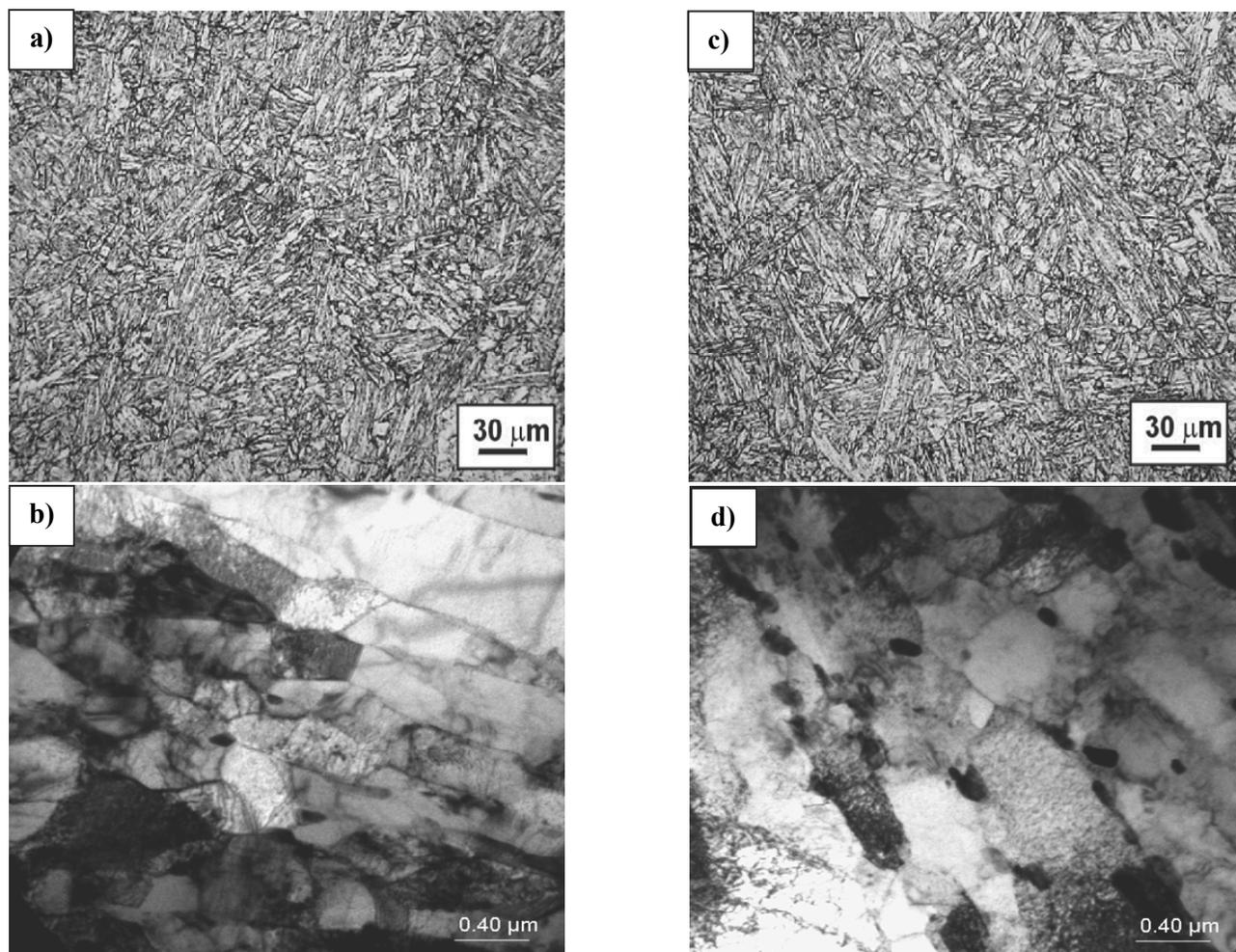


Fig. 6. Microstructure of GP91 cast steel after the heat treatment: a, b) austenitizing 1040°C and tempering 730°C; c, d) austenitizing 1100°C and tempering 730°C; a, c) OM; b, d) TEM, thin foil

Table 3.
Mechanical properties of GP91 cast steel in cast-state condition at room temperature [15]

	YS, MPa	R _{p0.2} , MPa	El., %	RA, %	KV, J	HV30
GP91	644	506	20	58	94	232

Required mechanical properties in the as-received condition (Table 3) is what GP91 cast steel owes to the lath microstructure of low-carbon martensite (Fig. 2) and the related mechanisms of matrix strengthening. The coarse grain cast structure of GP91 cast steel, unfavorable in terms of properties, is subjected to refinement with martensite laths. Refinement of the microstructure leads to an increase in strengthening with grain boundaries, which is favorable not only for plastic properties but

also for strength properties. According to the data provided by the literature [16] strengthening with grain boundaries in the case of martensitic structure is dependent on the width of martensite laths and for the width of 0.36 μm (similar for the investigated cast steel) it amounts to 239.4 MPa. There are also other strengthening mechanisms which play a significant role here and they are used in martensitic steels/cast steels for obtaining high mechanical properties, such as solution strengthening with atoms of alloy elements.

Table 4 includes the results of tests carried out on mechanical properties of GP91 cast steel in the as-cast state at the temperature of 550 and 600°C. Performed research at elevated temperatures has proved that the yield strength as well as tensile strength of the GP91 cast steel in the as-cast condition were higher than the minimum requirements. At the research temperature of 550°C the tensile strength was higher than the required minimum by 14%, and the yield strength - by 24%. At the temperature of 600°C these values were higher by 16 and 20%, respectively.

Table 4.
Mechanical properties of GP91 cast steel in cast-state condition at elevated temperatures

Temp. °C	YS MPa	R _{p0.2} MPa	El. %	RA %	min. YS ^t MPa	min. R _{p0.2} ^t MPa
550	375	334	13,3	60	330	270
600	302	259	18,7	76	260	215

* according to [12]

4. Structure and properties of GP91 cast steel after heat treatment

Heat treatment (hardening and high-temperature tempering) of GP91 cast steel allowed to obtain a fine-grained microstructure of high-temperature tempered martensite, regardless of the heat treatment parameters. Examples of the GP91 cast steel microstructure after the heat treatment are illustrated in Fig. 6. Austenitizing of GP91 cast steel within the temperature range of 1040-1100°C has contributed to the austenite grain size reduction in comparison with the as-cast state. As a result, it influenced the obtainment of a fine-grained martensitic structure after hardening.

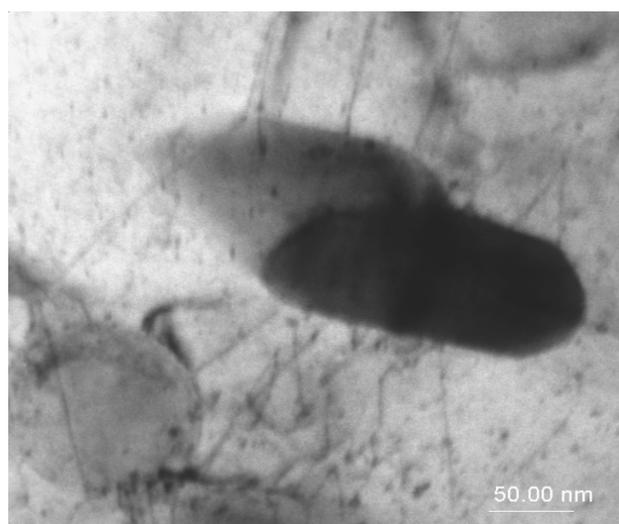


Fig. 7. Precipitation of $M_{23}C_6$ carbide on the boundary of martensite lath, visible fine-dispersion precipitates of MX inside the lath, TEM, thin foil

High temperatures of tempering and long holding times contribute to the precipitation of numerous carbides of diverse size. Precipitates of this type are observable not only on the boundaries of former austenite grains, but also on the boundaries

of martensite laths, and inside martensite grains. Additionally, high temperatures of tempering lead to the intensification of the processes of recovery and recrystallization of matrix, which manifests itself in the fall of dislocation density and an increase in the width of martensite laths, as well as their disappearance in favour of polygonized ferrite grains. However, long holding times at high tempering temperatures, as practice shows [2,4,6], are essential for the obtainment of a very stable microstructure of the casts designed for long-term service at elevated temperatures.

Grain size reduction has a favorable effect on mechanical properties of the cast steel in two opposite directions: at the same time it raises the yield strength and impact strength and decreases the nil ductility transition temperature.

Microstructures of GP91 cast steel after different variants of heat treatment were similar and they were characterized by a lath substructure of martensite with large dislocation density, as well as by polygonal ferrite grains. Between the martensite laths and between the subgrains there were dislocation boundaries occurring. Performed identifications of precipitates in GP91 cast steel after various heat treatment variants have revealed the presence of two precipitation types in the microstructure-carbides of $M_{23}C_6$ as well as nitrides and carbonitrides of MX (NbC, VX).

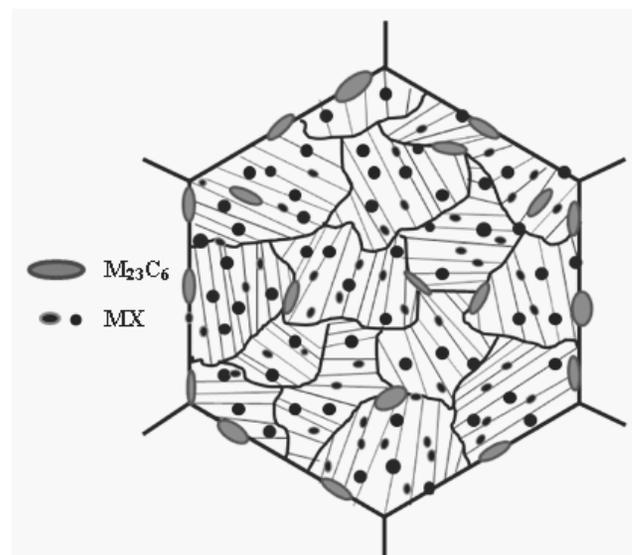


Fig. 8. Schematic illustrations of precipitation morphology in GP91 cast steel after heat treatment

Large number of $M_{23}C_6$ carbides of diverse size were located most of all on the boundaries of former austenite grain and on the boundaries of martensite subgrains/laths. Only few of the carbides of this type were seen also inside the ferrite subgrains. Fine-dispersive precipitates of the MX type, i.e. NbC carbides, as well as VX nitrides (carbonitrides) were observed on the dislocations inside the subgrains and on the subgrain boundaries (Fig. 7). Single precipitates of MX were also revealed on the boundaries of initial austenite.

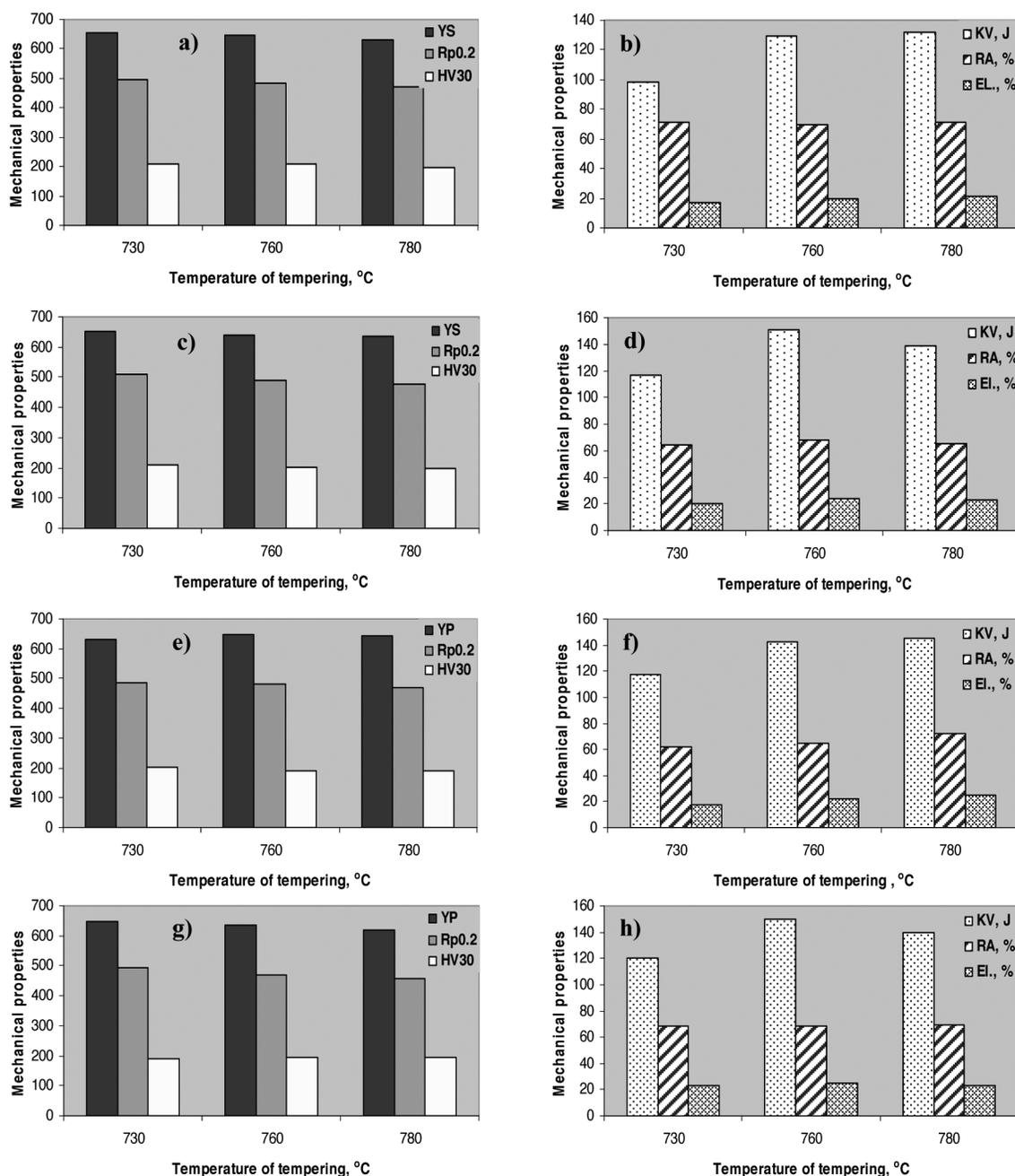


Fig. 9. Influence of the austenitizing temperature of: a, b) 1010°C; c, d) 1040°C; e, f) 1070°C; g, h) 1100°C on mechanical properties: a, c, e f, g); plastic properties: b, d, f, h) of GP91 cast steel after tempering at the temperature of 730, 760 and 780°C

Mean diameters of precipitates, depending on the heat treatment parameters, ranged from 97 to 159 nm for $M_{23}C_6$ carbides, while for the precipitates of MX—from 18 to 25 nm. The carbides of $M_{23}C_6$ precipitated on martensite subgrain/lath boundaries are of great importance because they stabilize the subgrain microstructure of martensite inhibiting the movement of dislocation boundaries.

The fine-dispersive precipitates of MX, precipitated on the dislocations inside martensite laths, provide high creep resistance by anchoring and inhibiting the dislocation movement. Fig. 8. presents the schematic view of morphology of precipitates in GP91 cast steel after heat treatment.

5. Mechanical properties of GP91 cast steel after heat treatment

Influence of heat treatment on mechanical properties of GP91 cast steel is presented in Fig. 9. The research performed on mechanical properties of GP91 cast steel after the heat treatment has proved that:

- heat treatment of GP91 cast steel allowed to obtain the standard required properties regardless of the assumed parameters of temperature and time;
- the highest strength properties (YS, $R_{p0.2}$, HV30) are provided (independently of the tempering temperature) by hardening from the austenitizing temperature of 1010 and 1040°C. Strength properties - $R_{p0.2}$ and HV30, insignificantly higher by around 3%, were obtained for the austenitizing temperature of 1040°C. The highest plastic properties, especially impact strength and elongation (KV and EL), were obtained after hardening from the temperature of 1100°C;
- after cooling from the austenitizing temperature of 1070°C it can be noticed that the strength properties (YS and TS) and plastic properties (El.) are insignificantly lower in comparison with the ones obtained for the lowest (1010°C) as well as the higher (1100°C) temperature of austenitizing;
- increasing the temperature of tempering from 730 to 780°C results in a reduction in strength properties by about 2-5%, depending on the temperature of austenitizing, and an increase in plastic properties: elongation by 15-39% and impact strength by 17-39%.

6. Influence of annealing after heat treatment on the microstructure and properties of GP91 cast steel

It may occur that after heat treatment of the steel casts there are technological damages appearing on their surface in the form of cracks and deformations. Such damages are removed mechanically. The defects are fixed through welding with electrodes with the chemical composition of weld metal being similar to the chemical composition of the cast material. After welding the steel castings are subjected to heat treatment in order to remove welding stresses-stress relief annealing [10,17]. Microstructure of GP91 cast steel after heat-toughening and stress relief annealing are shown in Fig. 10. After annealing GP91 cast steel was characterized by a microstructure similar to that of heat-toughened condition, i.e. microstructure of high-temperature tempered martensite with numerous precipitations.

The areas observed in the microstructure, similarly as in the heat-toughened condition, were the areas of lath structure characterized by large density of dislocations, as well as the areas of polygonized ferrite. Moreover, like in the heat-toughened condition, in the cast steel after additional treatment the occurrence of two types of precipitates was revealed: $M_{23}C_6$ carbides and precipitations of nitrides and carbonitrides of the MX type. After annealing at the temperature of 750°C the mean

diameter of $M_{23}C_6$ carbides amounted to 142 nm. The stress relief annealing, however, did not affect the size of MX precipitates in a significant way. Still, it should be expected that annealing the examined cast steel additionally for many hours at the temperatures above 700°C may lead to a reduction in its:

- dislocation strengthening as a result of the fall in dislocation density due to the processes of recovery and recrystallization of the matrix,
- solid solution strengthening by alloying elements as a result of diffusion of atoms of the carbide - forming elements (chromium and molybdenum) to the carbides of $M_{23}C_6$ type mainly and the related process of their coagulation, which leads to the decrease in precipitation strengthening.

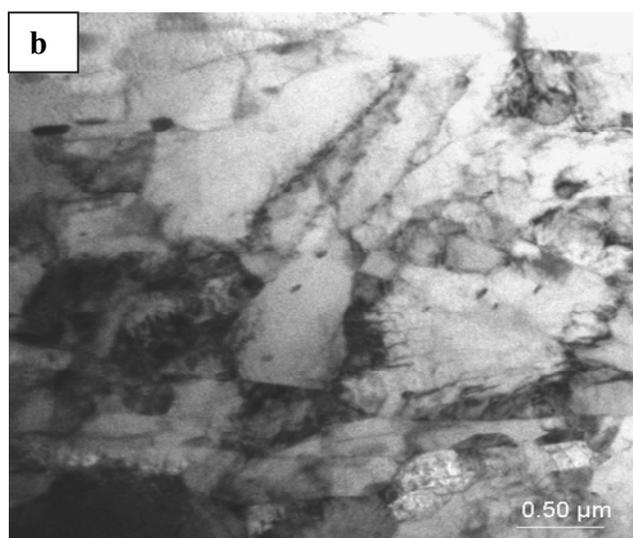
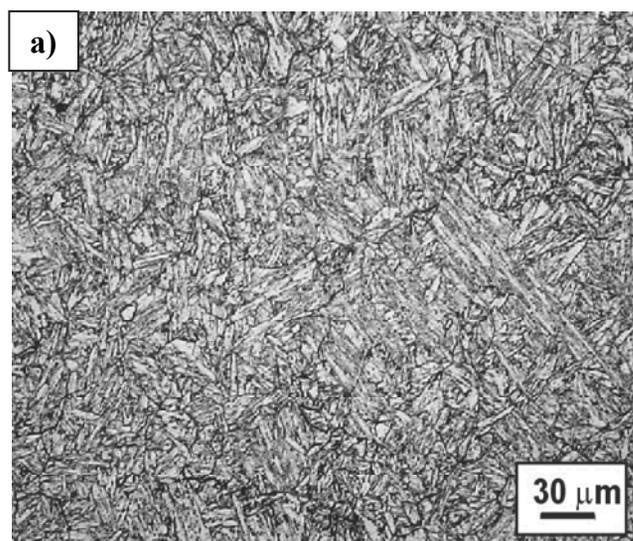


Fig. 10. Microstructure of GP91 cast steel after heat-toughening and stress relief annealing at the temperature of 750°C, a) OM; b) TEM, thin foil

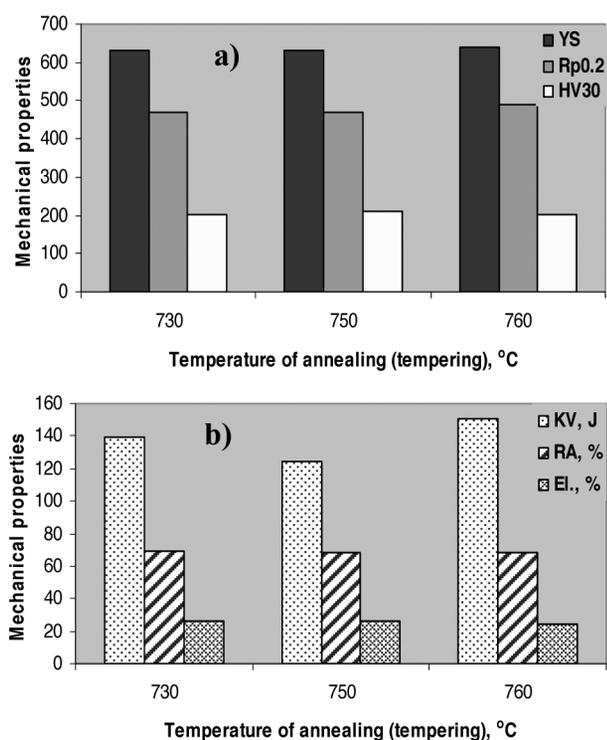


Fig. 11. Comparison of: a) strength properties; b) plastic properties of GP91 cast steel in the heat-toughened condition and GP91 cast steel after the additional heat treatment- stress relief annealing

Results of research on mechanical properties of GP91 cast steel after heat treatment with additional annealing are illustrated graphically in Fig. 11. The examined cast steel after the above-mentioned heat treatment met all standard required properties.

Holding at the temperatures of 730 and 750°C caused a slight reduction in strength properties of about 2-5% in comparison with the heat-toughened condition. Plastic properties of the investigated cast steel practically did not change. Similar phenomenon of a decrease in the yield strength and tensile strength of high-chromium cast steels with the extended time of tempering/annealing has been described in paper [9,10]. Reduction in the strength properties after annealing should be associated with the processes going on in the microstructure of the investigated cast steel, i.e.: decrease in the dislocation density, fall of alloy elements concentration in the matrix and growth of the precipitates size.

7. Conclusions

1. The GP91 cast steel in the as-cast state is characterized by a coarse grain microstructure of high mechanical properties which result from the lath microstructure of low-carbon martensite. High properties of GP91 cast steel in the as-cast condition indicate the dominant role in the mechanism of strengthening martensitic steels/cast steels, that is the role of

the dislocation boundaries between martensite laths, as well as the little significance of high-angle boundaries of former austenite grains in this mechanism.

2. Independently of the heat treatment parameters, GP91 cast steel in the heat-toughened condition has got a fine-grained microstructure of high-temperature tempered martensite with numerous precipitations of $M_{23}C_6$ carbides and nitrides, carbonitrides of MX. The areas occurring in the microstructure are: those with lath-like structure characterized by a large dislocation density, as well as those of polygonized ferrite.
3. Heat treatment of GP91 cast steel contributes most of all to an increase in plastic properties, mainly impact energy and reduction of area. Strength properties after the treatment are on the level of the properties of a cast steel in the as-cast condition, due to long holding times at the temperatures above 700°C.
4. Temperatures of austenitizing up to 1040°C seem to qualify as an appropriate hardening temperature for GP91 cast steel, as they provide high strength properties and impact energy $KV > 100$ J after tempering.
5. The highest strength properties for GP91 cast steel, regardless of the tempering temperature, are achieved after hardening from the austenitizing temperature of 1040°C. Whilst higher plastic properties are obtained for the tempered GP91 cast steel after hardening from the temperature of 1100°C. This allows to develop an optimum microstructure and properties through the selection of heat treatment parameters.
6. Heat treatment, simulating the stress relief annealing after the fixing of the cast through welding, contributes to a slight decrease in strength properties (on the level of 2-5%) with practically unchanged plastic properties. This proves high stability of the microstructure of the examined cast steel.

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