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INFLUENCE OF NITROGEN AND COPPER ON HARDENING OF AUSTENITIC CHROMIUM-NICKEL-MANGANESE STAINLESS STEEL

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ABSTRACT

Alloying by nitrogen has a strong effect on the whole complex of steel properties. Joint alloying by nitrogen and copper increases apparently austenite stability and decreases the beginning temperature of martensite transformation. The steels alloyed by copper and nitrogen also elevate their resistance to pitting and microbiological corrosion. The conducted investigation was based on laboratory steel melting with pure charge materials. Mechanical properties have been determined during testings for single-axial static extension at the temperature values +100 °C, +20 °C, -100 °C, -175 °C, and for impact bending at the temperature values +100 °C, +20 °C, -100 °C, -196 °C. Presence of ferromagnetic phase has been defined after testings via magnetometrical method. Equilibrium phase diagrams of the investigated steels have been calculated using Thermo-Calc program, critical nitrogen concentration values N_{KP} excluding forming of nitrogen bubbles during crystallization have been determined, as well as temperature values M_H , M_{D30} of the beginning of martensite transformation and parameter of pitting corrosion resistance (PREN). The results of testings at different temperatures have been visualized and analyzed in the kinds of deformation diagrams, non-magnetic properties, low temperatures of the beginning of martensite transformation M_H , M_{D30} . It is concluded based on these results that austenite of steels Cr19Mn10Ni6Mo2N is stable in thermal and deformation conditions at rather high deformation degree. Additional alloying by copper (up to 2%) of nitrogen steel Cr19Mn10Ni6Mo2N has a small effect on its mechanical properties, but increases its austenite stability (via lowering of the temperatures of the beginning of martensite transformation M_H , M_{D30}), eliminates forming of δ -ferrite in crystallization and rises pitting corrosion resistance, increasing PREN parameter. Austenite of classic stainless steels Cr18Ni9N and Cr18Ni9 has insufficient mechanical stability at negative temperatures, thereby these steels can be used for non-deformed products at the temperatures below -70 °C, because forming of deformation martensite in these steels is possible.

Key words:

austenitic stainless steels, alloying by nitrogen and copper, treatment, thermal stability, structure, mechanical properties, martensite transformation, ferromagnetic phase, pitting corrosion resistance

1. Introduction

Stainless steels have wide range of application practically in all industrial and economic branches. At the same time, the requirements to universalization of applied steels and their production technologies are permanently growing.

The stainless austenitic chromium-nickel steel 18Cr-10Ni has been created in 1912 and remains the classic basic alloying composition for austenitic stainless steels. The existing contradictions between corrosion resistance, mechanical properties, workability and cost of material and finished products have supported the continuous improvement of steel compositions and their fabrication and processing methods.

The main tendency in steel cheapening is still lowering of nickel content via its replacement by manganese that is also a γ -stabilizer. However, alloying by manganese decreases resistance to pitting corrosion in sea water and in $H_2SO_4 + NaCl$ mix [1], while this resistance is critical for stainless steels. Forming of MnS inclusions that are the most preferable area of artificial pits among all inclusions can be considered as one of the causes of this decrease pitting corrosion resistance. It is connected with the fact that all inclusions in stainless steels have less value of the coefficient of thermal expansion compared with austenite, thereby appearing tensile stresses facilitate tearing of the forming protective passive film. To increase corrosion resistance, chromium, nickel and molybdenum content is

increased and sulfur content is decreased, i.e. steel purity for inclusions is improved.

The negative effect of manganese can be compensated by steel alloying by nitrogen [2, 3]. Even small nitrogen addition increases substantially the potential of pitting forming, shifting it in the area of positive values and expanding the area of passive potential. Positive nitrogen effect on resistance to pitting corrosion and other kinds of corrosion mostly manifests in combination with molybdenum [1, 4]. In order to express the links between alloying elements and pitting corrosion resistance in the solution of NaCl and $FeCl_3$ chlorides, the parameter names numeric equivalent of pitting corrosion resistance (PREN) has been formulated on the base of experimental investigations of large number of stainless steels [4]:

$$PREN = Cr + 3.3Mo + 36N + 7(Mo)(N) - 1.6Mn. \quad (1)$$

At a first approximation it is concluded that the more is PREN parameter, the more is the value of pitting corrosion resistance. The equation (1) testifies that molybdenum and nitrogen additions are more effectively for increase of pitting corrosion resistance of stainless steels than chromium addition by 3 and 36 times respectively; interaction between nitrogen and molybdenum are also a significant factor. The PREN calculation formula didn't take into account the copper influence, though the results of experimental tests have testified that copper addition has led to increase of pitting forming potential in

chloride-containing media and to more slow transpassive dissolution of salts [1].

Additions of copper and jointly copper and nitrogen increases essentially austenite stability and lowers M_H [5] and M_{d30} [6]:

$$M_H = 500 - 25(\text{Ni} + 0.8\text{Cr} + 1.2\text{Mn} - 8) - 12.5\text{Mo} - 29.4\text{Ti} - 35\text{Nb} - 32.6\text{Cu} - 8.4\text{Co} - 25.4(\text{C} + \text{N}), \quad (2)$$

$$M_{d30} = 497 - 462(\text{C} + \text{N}) - 9.2\text{Si} - 8.1\text{Mn} - 13.7\text{Cr} - 20\text{Ni} - 18.5\text{Mo}. \quad (3)$$

It allows to expand significantly application of such steels as non-magnetic cryogenic materials in the area of low temperatures and high loads.

Peculiar influence of manganese on such physical properties as magnetic permeability at cryogenic temperatures (4.2 K) and coefficient of thermal expansion should be mentioned especially. According to [7], low-nickel (1–5% Ni) medium-manganese (9–15% Mn) stainless steels containing 17–20% Cr can be considered as well-used and prospective material for further development of components and constructions in superconductive hadron collider (CERN).

Peculiar properties of copper manifest, in particular, in appearing biocidity effect in copper-alloyed steels and in increasing microbiological corrosion resistance in steels alloyed by copper and nitrogen [8, 9].

Nitrogen is a prospective alloying element, at present time it is widely used in steels of different grades [2, 10, 11]. Nitrogen as well as carbon is an interstitial element and participates in forming of excessive phases. Nitrogen influence on steel properties is other than carbon influence. E.g., nitrogen unlike carbon increases not only steel strength, but also steel corrosion resistance. Nitrogen provides steel hardening via two mechanisms: as an interstitial element in solid solution and through precipitation of nitrides. The main nitrogen value is the fact that it is an effective austenite stabilizer and allows to decrease nickel content in steel. In general nitrogen alloying has a strong effect on the mechanisms of deformation and hardening and on the whole complex of steel properties [12–14].

Development of nitrogen steels during recent decades and proper approach to choosing nitrogen content and methods of its introduction, as well as to appointment of correct procedures of heating and heat treatment taking into account the processes of precipitation of complicated nitrides and carbonitrides, has been observed. Indeed, it has given a “second breath” to application of high-manganese steels, to development of chromium-manganese and especially manganese high-strength steels. Addition of manganese increased nitrogen solubility in liquid metal and in solid solution, as well as critical nitrogen concentration (N_{KP}), i.e. concentration when nitrogen bubbles are not forming during steel crystallization [15]. It allows to increase ni-

trogen content in steel and degree of its influence on metal structure and properties. Additional alloying by copper, together with usual hardening (with its efficiency close to hardening by manganese [6]) makes it possible to get the special properties of stable non-magnetic austenitic steels.

The aim of this work was examination of complex alloying by nitrogen and copper for hardening of chromium-nickel-manganese austenite stainless steel in wide temperature range.

2. Methods of the research

Chromium-nickel-manganese steels of Cr19Mn10Ni6Mo2N grade, alloyed by nitrogen (1A–3A) and additionally by copper (2A, 3A) and steels of Cr18Ni9 grade with different nitrogen content (5A, 6A) have been investigated. The chemical composition of these steels is presented in the **tab. 1**.

Steel melting has been conducted in the vacuum induction furnace with pure charge materials. Iron, nickel and molybdenum have been loaded in the furnace crucible. Heating and melting of charge materials have been conducted in vacuum. After complete melting of charge materials and termination of gas extraction argon (up to 1 atmosphere) has been introduced in the furnace and afterwards melting has continued in the argon atmosphere. Introduction of alloying components has been undertaken in the following sequence: chromium, copper, deoxidizers (aluminium and silicon), manganese, nitrogenized ferrochromium. The amount of activated ferrochromium has been determined by the required nitrogen content in steel, taking into account its 75% absorption. Nitrogen in the argon atmosphere is desorbing from the melt, thereby operation time after addition on nitrogenized ferroalloys and until the end of melting has been kept minimal and constant. It was necessary for decrease of nitrogen losses and assured reaching of the required content for nitrogen.

Metal has been cast in cylindrical moulds with feeder head. Ingot mass made 9.5 kg, and after cutting the feeder head it made 7.5 kg, while its dimensions were as follows: diameter 80 mm and height 170 mm. Ingots have been subjected to hot forging and hot rolling to thickness 8 mm and 6 mm for consequent extension tests and impact bending tests respectively. Temperature of metal forming corresponds to austenite area and temperature of the end of rolling process correlates with the temperature of processing for solid solution with water cooling.

Mechanical properties have been determined during tests for single-axial static extension and impact bending. Extension tests have been conducted using

Table 1. Chemical composition of the experimental steels

Steel marking	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	N
1A	0.096	0.232	10.12	0.011	<0.005	18.72	1.681	6.129	0.014	0.311
2A	0.092	0.198	10.10	0.014	<0.005	19.20	1.689	6.001	2.048	0.354
3A	0.078	0.210	10.05	0.014	<0.005	18.85	1.651	5.850	1.950	0.362
5A	0.035	0.199	1.238	0.014	0.011	18.54	0.046	8.633	0.0064	0.020
6A	0.043	0.221	1.322	0.014	0.009	18.43	0.045	8.883	0.011	0.180

“INSTRON-3369” unit at the temperature values +100 °C, +20 °C, –100 °C, –175 °C (–163 °C for steel 6A), with loading rate 2–10 mm/min according to the GOST 9651–84, GOST 1497–84 and GOST 22706–77 respectively. Impact bending test have been conducted using impact testing machine “Roell Amsler” on the samples with U-type concentrator, at the temperature values +100 °C, +20 °C, –100 °C, –196 °C in accordance to GOST 9454–78.

For extension testing at +100 °C, a sample has been heated in the temperature chamber of “INSTRON-3119-409” unit, for impact bending testing a sample has been heated in the tank with boiling salty water. For testing at negative temperatures, nitrogen vapours have been used at the temperature –100 °C, liquid nitrogen – at more low temperatures. For cooling of samples and keeping the required temperature cryostat was used.

Presence of ferromagnetic phase has been determined via magnetometrical method using eddy current flow detector MBП-2M having the range of ferrite phase measurements from 0.1 to 80%.

3. Results of the research

Mechanical properties of the experimental steels determined at different test temperatures are presented in the **tab. 2**.

The results of extension tests of the samples have shown that high level of strength properties has been achieved for steels 1A, 2A and 3A at room temperature, in comparison with conventional stainless steel Cr18Ni9 (5A), including nitrogen-alloyed steel (6A). Strength and plasticity for the steels alloyed by nitrogen and copper (2A and 3A) as well as steel 1A are practically equal.

Significant increase of tensile strength and yield point – by 1.4 times for the steel 2A and by 1.6 times for the steels 1A and 3A is observed at the test temperature –100 °C, comparing with the data obtained at room temperature. More essential strength elevation, by 2 times in average, is observed during testings at –175 °C. At the same time

these steels are also characterized by increase of relative narrowing (from 56–62% at room temperature to 83–84% at negative temperatures) and elongation (from 27–33% at room temperature to 45–50% at –100 °C and to 33–40% at –175 °C). Relative elongation for the steel 6A at –163 °C is essentially lower than at 20 °C. Tests of the steels 1A, 2A and 3A at +100 °C in the most cases manifested slight variation of strength and plasticity (see table 2).

The steels 1A, 2A and 3A remained to be non-magnetic (with content of ferromagnetic phase less than 0.1%) after extension tests at positive temperatures. Samples of the steels 2A and 3A after tests at –175 °C and part of samples of the steel 3A at –100 °C became weak-magnetic in the neck, while samples of the steel 1A became magnetic in the neck after testing at both negative temperatures. The steels 5A and 6A tested at +20 °C were weak-magnetic for $\delta \geq 60\%$ and $\psi \geq 70\%$, while the steel 6A was strong-magnetic after testing at –163 °C. Thereby austenite of the steels 1A, 2A and 3A is stable for deformation for δ up to 25–30% and ψ up to 50–70% at the temperatures from +20 till +100 °C, while austenite of the steels 5A and 6A has insufficient mechanical stability for $\delta \geq 60\%$ and $\psi \geq 70\%$.

The results of impact bending tests have displayed that the examined steels are characterized by rather high impact toughness for corresponding testing temperatures, and its value decrease with lowering of testing temperature. So, the following parameters have been obtained in average for the steels 2A and 3A: at room temperature $KCU = 2,53 \text{ MJ/m}^2$, $KCU_{-100} = 2,05 \text{ MJ/m}^2$, $KCU_{-196} = 1,35 \text{ MJ/m}^2$. Samples of the steel 6A have displayed more high level of impact toughness comparing with the steels 1A, 2A and 3A, owing to less values of strength and austenite stability due to realization of TRIP effect in the area of plastic deformation. However, these samples of the steel 6A have remained to be non-magnetic after impact bending tests at negative and positive temperatures (from –175 to +100 °C).

Strength and plasticity properties during extension of the steels 2A and 3A alloyed by nitrogen and copper don't differ practically from properties of the steel 1A that is alloyed only by nitrogen. Impact strength of these steels is a little lower than that of the steel 1A at all testing temperatures.

4. Discussion about the results

All investigated steels are austenitic at room temperature, because they are located in austenite area on the Schaeffler diagram (**fig. 1**).

Equilibrium phase diagrams have phase composition of the steels in the temperature area above 900 °C rather close to the real one and are valid for determination of heating temperatures before hot deformation and during quenching. To determine the ageing conditions, it is necessary to attract additionally the data about transformation kinetics and extraction of phases, because the processes of transformation and extraction of excessive phases don't reach equilibrium state at steel heating up to 500–600 °C during technologically permissible time. **Fig. 2** presents the polythermic sections of state diagrams

Table 2. Mechanical properties of the investigated steels

Steel marking	$T_{\text{test}}, ^\circ\text{C}$	σ_B, MPa	$\sigma_{0,2}, \text{MPa}$	$\delta, \%$	$\psi, \%$	KCU, MJ/m ²
1A	+100	970	615	25	52	3.15
	+20	925	690	33	62	2.90
	–100	1410	1140	47	83	2.25
	–175 (–196)*	1775	1405	33	84	1.55
2A	+100	910	625	27	55	2.55
	+20	940	700	27	56	2.50
	–100	1285	970	45	84	2.00
	–175 (–196)	1825	1440	40	84	1.35
3A	+100	865	610	33	69	2.65
	+20	915	645	31	58	2.55
	–100	1355	1055	50	84	2.10
	–175 (–196)	1535	1480	37	84	1.35
5A	+20	570	250	60	70	-
6A	+20	750	400	63	84	3.90
	–163	1065	645	39	78	2.50

* extension tests at –175 °C, impact bending tests at –196 °C.

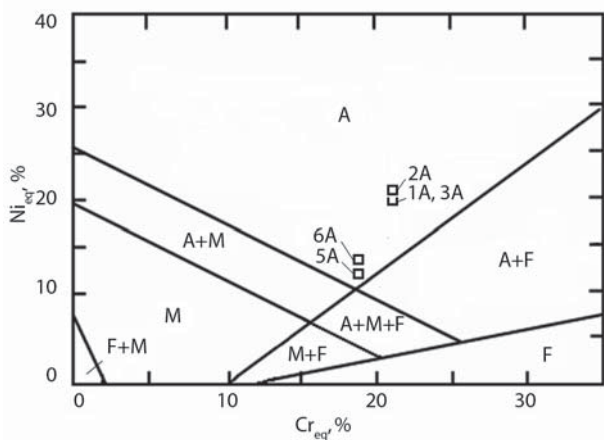


Fig. 1. Location of the examined steels on the Shaeffler diagram

for investigated steels at variable nitrogen content and at constant content of other alloying elements. The diagrams are built in the program Thermo-Calc on thermodynamic base TCFE6. Calculation in the program Thermo-Calc has been conducted via CALPHAD method the program Thermo-Calc. One common diagram is built for each of the steel pairs: 2A and 3A, 5A and 6A, because they have close chemical composition.

Vertical lines on the fig. 2 correspond the actual nitrogen content in steels. Based on the equilibrium diagrams, the temperature areas of solid solutions γ , while critical nitrogen concentrations N_{KP} (see **tab. 3**) have been determined via the method [15]. This table also presents the results of calculations of M_H , M_{d30} and PREN for the examined steels according to the formulas (1)–(3).

The diagrams testify that crystallization of the steels 5A and 6A is finishing by forming of dual-phase structure of solid solutions $\delta+\gamma$. In this case the steel 5A, i.e. usual stainless steel Cr18Ni9 has the most wide temperature area $\delta+\gamma$ (from T_{sol} to 1230 °C). Nitrogen addition in this steel decreases the area of $\delta+\gamma$ solutions, but increases the lower temperature of γ -area down to 1000 °C, and forces to rise the steel heating temperature before thermal deformation processing. Nitrogen content in this steel can't be higher than 0.20% because the correlation $N < N_{KP}$ should be valid for elimination of forming of nitrogen bubbles, owing to extraction during δ -phase

Table 3. Estimations of N_{KP} , temperatures of M_H , M_{d30} and PREN as well as temperature intervals in γ -area of the examined steels

Steel marking	$N_{KP}\%$	Boundaries of γ -area, °C			M_H , °C	M_{d30} , °C	PREN
		T_{max}	T_{min}	$\gamma + AlN$			
1A	0.49	1237	1120	950	-256	-185	22.9
2A	0.38	T_{con}	1160	1105	-338	-207	25.5
3A	0.38	T_{con}	1160	1105	-321	-195	25.4
5A	0.26	1230	890	–	61	32	17.4
6A	0.26	1370	1000	930	12	-50	23.0

crystallization and connected with this process nitrogen segregation. Thereby the achieved values of solid solution hardening of this steel caused by nitrogen ($\sigma_B = 750$ MPa, $\sigma_{0.2} = 400$ MPa, see **tab. 2**) are maximal possible.

Crystallization of the steel 1A usually finalizes in forming of two $\delta+\gamma$ solutions existing in the temperature area from T_{sol} up to 1237 °C. This steel has also rather wide area of γ -solutions (see **tab. 3**). If we don't take into account AlN influence, the lower boundary of γ -area equals to 950 °C, i.e. close to this boundary of the steel 6A. Al content in calculations is accepted on the base of the results of chemical analysis. Practically whole aluminium is connected with oxygen as Al_2O_3 oxides and it is possible not to take it into account. Critical nitrogen concentration for the steel 1A makes 0.49%. Thereby nitrogen concentration in this steel can be increased up to 0.40% without danger of forming of nitrogen bubbles and, if required, it is possible to provide higher hardening in comparison with the data in **tab. 2**. In this case the lower boundary of γ -area (more exactly to speak about $\gamma + AlN$) increases only up to 1000 °C, what is quite acceptable from the technological point of view. Comparison of the diagrams show that the steel 1A is characterized by lower temperature of $\gamma \rightarrow \alpha + \gamma$ transformation (560 °C) compared with the steels 5A, 6A (650 °C), thereby by more stable austenite at increased temperatures.

Copper decreases nitrogen solubility in steel both in liquid and solid states ($N_{KP} = 0.38$). Additional alloying of the steel 1A by copper up to 2% has not practical effect on its strength and plasticity at all testing temperatures (see **tab. 2**). Comparison of the diagrams on the **fig. 2, b and c** testify that copper addition leads to shift of γ -area to the solidus temperature. Crystallization of the steels 2A and 3A is conduct-

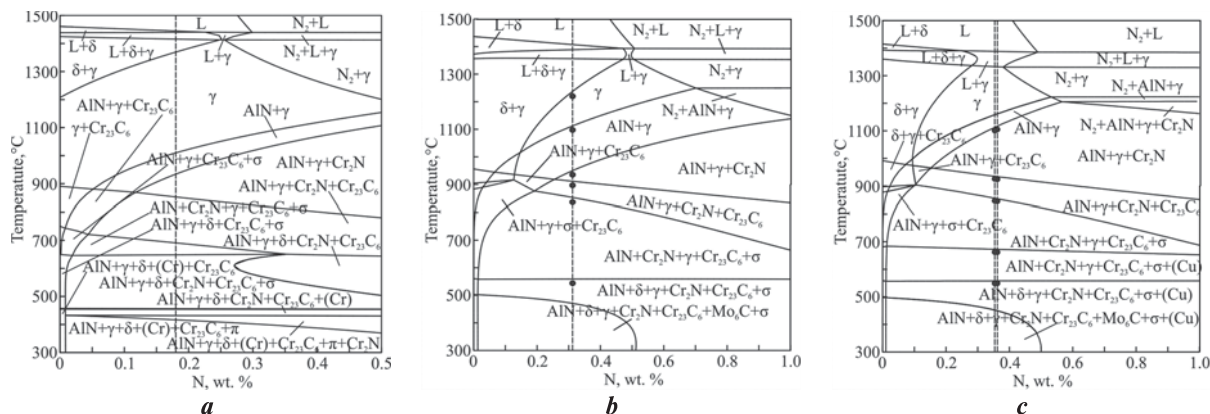


Fig. 2. Polythermic sections of the state diagrams for the steels 1A-6A with variable nitrogen content: a – 5A, 6A; b – 1A, c – 2A, 3A

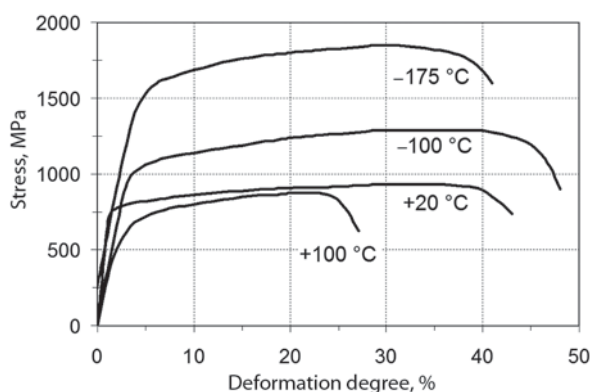


Fig. 3. Deformation diagrams after extension tests at different temperatures for the steel Cr19Mn10Ni6Mo2Cu2N (2A)

ing via the route $L \rightarrow L + \gamma \rightarrow \gamma$, i.e. δ -ferrite is not forming and this is a positive factor. However, in this case the lower boundary of γ - and $\gamma + \text{AlN}$ areas increases up to 1108 °C, what requires higher steel heating temperatures for thermal deformation processing comparing with the steel 1A. This temperature can be decreased down to 1000 °C via lowering of nitrogen content to 0.2%, but it is accompanied by decrease of steel hardening degree. Fig 2, *c* shows that addition of 2% of copper has no effect on the temperature of $\gamma \rightarrow \alpha + \gamma$ transformation for the steel 1A.

Deformation diagrams of the steels 1A, 2A and 3A are smooth and don't display any signs of ageing and phase transformations during extension tests at the temperatures from –175 to +100 °C (**fig. 3**).

The results of the series of tests conducted at the temperatures from +100 °C to –196 (–175) °C have been analyzed for deformation diagrams, non-magnetic properties, low temperatures of the beginning of martensite transformation M_H and M_{d30} . It can be concluded that austenite in the steels 1A, 2A and 3A is characterized by thermal and deformation stability at rather high deformation degree. Additional alloying of the nitrogen steel 1A by copper increases austenite stability, lowers the temperatures of the beginning of martensite transformation M_H and M_{d30} , excludes forming of δ -ferrite during crystallization and rises pitting corrosion resistance with elevation of PREN parameter (see tab. 3). Austenite of the steels 5A and 6A has insufficient mechanical stability at negative temperatures, forming of deformation martensite is possible in these steels, that's why they can be used for non-deformable products at the temperatures below –70 °C.

5. Conclusions

Nitrogen being in the solid solution as an interstition element allows to achieve significant hardening with reserve of plasticity and toughness, while the steel remains in austenitic state. Solid-solution hardening of chromium-manganese austenitic steels by nitrogen also increases their pitting corrosion resistance in chloride-containing media.

Low-nickel corrosion-resistant steels 1A, 2A and 3A can be used at the temperatures from +100 to –175 °C; in

these conditions such steels have stable austenitic structure and high mechanical properties. The steels 1A, 2A and 3A have higher strength in comparison with conventional corrosion-resistant steel 5A; it is provided owing to solid-solution hardening, due to higher alloying degree (alloying by nitrogen). Consequent processing in the technological route of these steels should exclude holding in dual-phase areas to prevent precipitation of carbides and nitrides.

Complex alloying of chromium-manganese steels by nitrogen and copper is efficient for hardening, rise of thermal and mechanical stability of austenite, its corrosion resistance as well as for achievement of the new consumer's properties of steels.

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