Effect of alloying elements on structure, metastability and properties of Fe-Cr-Mn (Ni-free) corrosion-resistant austenitic-ferritic steels

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Abstract. Investigated was chromium influence within 15-21.4 wt.% and manganese influence within 8-11.4 wt.% upon phase transformations, formation of structure and mechanical properties of corrosion resistant nickel-free (Fe-Cr-Mn) steels of austenite-ferrite structural class. It was found out that by the variation of chromium and manganese content it was possible to modify efficiently the content of ferrite and austenite in the structure, the degree of deformation metastability of austenite and control the mechanical properties of steels. An improved complex of strength, plasticity properties and impact toughness is reached at optimal content of alloying elements (Cr, Mn) in new nickel-free steels, it can be explained by positive impact of deformation induced martensite \( \gamma \rightarrow \alpha' \) transformation at testing during their formation.

1. Introduction

In the world practice, Cr-Ni steels of 18/8 (10Cr18Ni9Ti) type, austenite structural class, 08Cr22Ni6Mo2 austenitic-ferrite class, etc. are widely used as effective stainless (corrosion-resistant) steels. These steels are very expensive since they contain 6-11% Ni [1-3]. Therefore, in many countries research and development are being carried out on low-nickel and nickel-free steels capable of adequately replacing the above-mentioned Cr-Ni corrosion-resistant steels, retaining a sufficient set of mechanical properties and corrosion resistance [4-6].

With the objective of adequate replacement of expensive and scarce Fe-Cr-Ni corrosion-resistant steels, Fe-Cr-Mn (nickel-free) steels of austenitic-ferritic class have been developed for the ability to work in corrosive environments of low and medium aggressiveness including sea water [7]. However, the literary data on implementation of the deformation induced martensite transformation during the tests its role in formation their properties are scarce [7-10].

The objective of this work is to investigate the effect of chromium content and quenching parameters on the phase composition, the austenite metastability and properties of nickel-free corrosion-resistant steels of the austenitic-ferritic class.

2. Research and test methods

Variable correlations of alloying elements, mainly Mn, Cr, C, Si, were developed for smelting new alloyed steels with experimental compositions with the objective of obtaining different phase correlations of austenite and ferrite amounts in the structure of experimental steels. Steels were smelted in “IST-016” induction furnace, possessing basic chromium-magnesite refractory lining and then they were cast into shell moulds in the shape of blanks for specimens. Ferroalloys were used at smelting (low carbon ferro-chromium, ferro-silicon, ferro-molybdenum, as well as metallic manganese and copper, aluminium and
silica-calcium for deoxidation and refining of steel. Specimens for investigation and testing of mechanical properties were made from moulded blanks by means of machining and then they underwent the specified heat treatment modes. Chromium content in the smelted steels was varied within 15 – 21.4 wt.% limits, while manganese content within 8-11.4 wt.% in numerous combinations for obtaining different phase quantitative correlations between austenite and ferrite. Also investigated was standard 12Cr18Ni9Ti (18-10) chromium-nickel steel in hot rolled state.

Investigated were specimens of developed stainless steels on Fe-Cr-Mn basis, with chromium content varying within the specified limits. The specimens underwent quenching from 1,050 °C temperature into water. Investigation of microstructure of stainless steels specimens was carried out on an optical metallographic microscope “NEOPHOT-21” (manufactured by Carl Zeiss Firm) and “MMT14”, at magnifications of x100-400. The phase composition (amount of ferrite, austenite) was determined by x-ray method on diffractometer “DRON-3” in Feα-radiation.

Micro-hardness test of phase and structures composition was studied on micro-hardness tester “NOVOTEST TC-MKB-1” by pressing in a pyramidal indenter with an apex angle of 136° under a load of 50 gf. Hardness was determined by the Rockwell method on the B scale (HRB) with a load of 100 kgf (GOST 9013-59). Tensile tests were carried out on 5-fold samples with a diameter of the workpiece part of 6 mm (the estimated length of the workpiece was 30 mm) on a YMM–5 testing machine with a maximum load of 50 kN, tensile rate being 2 mm∙min\(^{-1}\) in accordance with GOST 1497-84 and ISO 6892-2015. Dynamic bending (impact strength) tests were performed on samples with a cross section of 10×10 mm, 55 mm length and U-shaped (R0.25 mm, depth 2 mm) on a “MK-30” impact testing machine with maximum potential energy of 300 J (GOST 9454-78).

3. Research results and discussion

The work presented the task solution of creating of new nickel-free stainless steels, based on the results of the previous investigations of such steel carried out at PSTU [7]. As the bulk of pump parts and other facilities operating in sea water are manufactured by means of casting, the objective was to create steels, obtained by smelting and cast part for subsequent exploitation in cast state following heat treatment.

The results of the previous investigations proved that it would be advisable to develop nickel-free and low-nickel steels of austenite-ferrite structural class on Fe-Cr-Mn basis. Therefore, for the development of the compositions of new steels, an alloying system Fe-Cr-Mn with a balanced content of chromium, manganese, carbon. The results of the previous investigations proved that it would be advisable to develop nickel-free and low-nickel steels of austenite-ferrite structural class on Fe-Cr-Mn basis. Therefore, for the development of the compositions of new steels, an alloying system Fe-Cr-Mn with a balanced content of chromium, manganese, carbon.

Alongside with these elements it would be advisable to alloy steels with small additions of silicon, copper, molybdenum or vanadium and nickel. The content of carbon, chromium and manganese should be strictly well-balanced in order to ensure austenite-ferrite phase composition with an opportunity to control the number of phases by selecting the above-mentioned alloying components and heat treatment modes. The carbon content should be as little as possible, not exceeding 0.12 weight % (hereinafter wt.%) to ensure high corrosion resistance, mechanical properties and weldability. Chromium and manganese content was planned to be varied within the specified parameters for adjusting the amount of austenite and ferrite, it having no effect upon the degree of deformation metastability of austenite. Silicon was introduced into steel with the objective of strengthening ferrite and austenite and also for improving heat and corrosion resistance in case new steels are applied not only as stainless but also as heat resistant.

Steels were alloyed with little additions of copper with the idea of improving plasticity, corrosion resistance and additional austenite stabilization. Molybdenum was introduced in small amounts for increasing corrosion resistance under the seawater conditions. Small nickel additions (less than 1 wt.%)
into chromium-manganese steels could be optional (recommended) for improving corrosion resistance and impact elasticity (especially at low temperatures). It is worthwhile mentioning here that according to the requirements of GOST 5632-2014 chromium-manganese corrosion resistant steel may have residual nickel mass share up to 2.0 wt.%, that is not alloying with nickel.

Developing new steels was based on the idea of creating a metastable state of the austenite component, which in the process of testing of mechanical properties and exploitation is capable of transforming into deformation martensite, due to deformation induced martensite $\gamma \rightarrow \alpha'$ transformation at testing (hereinafter DIMTT). Such transformation ensures additional and essential improvement of mechanical properties and even the entire complex of them, which cannot be realized on the well-known analogues - stainless (corrosion-resistance) chromium-nickel steels of the austenite class (steel 18-8 or 18-10 – AI304 (10Cr18Ni9Ti)) and austenite-ferrite class (8Cr22Ni6Mo2, GOST 5632-2014).

3.1 Effect of chromium and manganese content upon the microstructure of the developed and investigated corrosion-resistant Fe-Cr-Mn steels

The microstructure of the investigated cost-saving alloyed 2-phase (austenitic-ferritic) steels with different chromium and manganese content and small additions of copper, silicon, nickel and molybdenum in some of them is summarized in Figure 1. It represents polyhedral (oval-shaped) austenite grains with closed ferrite inclusions of irregular shape. Austenite grains are clearly visible, round-shaped, the ferrite component is represented as inter-layers between austenite grains. Also observed are dispersed inclusions chromium carbides particles $(\text{Cr,Fe})_23\text{C}_6$. Detachment of $\sigma$-phase on grain boundaries is not excluded when the chromium content grows.

Manganese is an austenite forming element and in our case with the content varying (8 to 11.4 wt.%) austenite is stabilized for transformation at cooling, having been introduced instead of costly and deficient nickel. As a result of heating up to 1,050 °C prior to quenching chromium carbides are mainly dissolved. Due to heating the amount of austenite is increased, the degree of stability of $\gamma$-phase growing at the same time. The increase of stability can also be explained by the growth of the degree solubility of carbides, their enrichment with carbon and chromium and, also, by possible redistribution of manganese content between ferrite and austenite at heating.

The microstructure of 10Cr15Mn10 steel (15.0 wt. % Cr) represents austenite grains of angular and elongated shape with ferrite and carbide inclusions. As a result of heat treatment (quenching at 1,050 °C) the austenite amount in the structure of steel increases (see Figure 1a).

The microstructure of 09Cr17Mn9 steel (16.59 wt. % Cr) represents an austenite matrix with dendritic austenite grains and ferrite zones around the austenite grains. The results of the x-ray structural phase analysis showed that austenite amount was austenite (A) = 52%, while ferrite (F) = 48% (Figure 1b). In the structure of 09Cr18Mn10 steel (17.26 wt. % Cr) austenite is partially twinned, deformation twins are observed, while some austenite grains reveal the signs of $\varepsilon'$- and $\alpha'$ – martensite’s. This is explained by deformation metastability, i.e. the capability of $\gamma \rightarrow \varepsilon'$ and $\gamma \rightarrow \alpha'$to DIMTT in the thin surface layer of microsection at grinding and polishing in the process of preparation of microsections (see Figure 1c). A tendency is observed towards an increase of ferrite content in steels with increased from 17 wt.% to 20 wt.% content of chromium, for instance for 09Cr17Mn9 steel (16.59 wt. % Cr) (Figure 1b) the content of F = 47%, A = 52%, and for steel grade 09Cr20Mn9 with 20 wt. % Cr (Fig. 4) F = 62%, A = 38%.

The microstructure of 09Cr20Mn10 steel (20.7 wt. % Cr) is not homogeneous, dendritic structure can be traced in dark grains. The content of the austenite phase is reduced (A=38%) while the ferrite phase increases (F=62%) it is explained by the influence of ferrite-generating chromium. 09Cr21Mn10 steel that has the biggest chromium content 20.6 wt.% the microstructure represents an austenite matrix with ferrite areas of irregular shape (see Figure 1d). Carbide inclusions (C) are also seen. According to the data of x-ray structural phase analysis that steel contains - 69% A and 31% F.
Figure 1. The microstructure of corrosion-resistant steels with different contain of chromium and manganese after quenching at temperature 1,050 °C: a) 10Cr15Mn10; b) 09Cr17Mn9; c) 09Cr18Mn10; d) 09Cr21Mn10; (A – austenite; F – ferrite; C – carbide).

Having analyzed the data mentioned above we may suppose that in 09Cr21Mn10 steel austenite microhardness is increased from 331-335 HV to 376 HV, it being explained by strengthening under the influence of chromium. However, austenite microhardness in 09Cr20Mn10 steel is just 316 HV at its smaller amount (A = 38%) as compared to other steels. It is quite obvious that chromium is distributed unevenly inside steels structure, especially between ferrite and austenite, accordingly ferrite microhardness is increased under the influence of chromium.

On the whole austenite grains possess a polyhedral (oval) shape, while in 10Cr15Mn10 steel or in 09Cr17Mn9 dendrite austenite grains are observed quite often. It is quite typical of the microstructure of cast metal. Particularly, in steels with the highest chromium content (20.7 wt.% 09Cr21Mn10 and 09Cr20Mn10, ferrite is observed as a branched matrix, which is typical of cast metal.

The microstructure of standard 12Cr18Ni10Ti steel (taken for a comparative analysis) (Fig. 6) mainly austenite, though presence of small δ-ferrite grains can’t be excluded, it being allowed by the requirements of GOST 5949-75.

The microhardness of chromium- nickel austenite - 278 HV is slightly lower than of chromium-manganese (320-386 HV), as it does not contain Mn, that strengthens austenite much more than Ni, accordingly, Cr – Mn austenite in the structure of new steels has greater microhardness - 320-386 HV.
Such difference in the value of austenite microhardness can be explained by existence of $\gamma \rightarrow \alpha$ transformations at heating for quenching under the influence of manganese (areas of existence of these phases can be different temperature-concentration fields) and the heating temperature 1,050 °C. These factors and heating conditions cause $A \rightarrow F$ or $F \rightarrow A$ transformations, ferrite and austenite are enriched with various alloying elements (Cr, Mn, Si, Cu, Mo) during holding during heating for quenching, it changing the correlation between ferrite and austenite [7, 11]. The structural transformations in austenite-ferrite steels are complicated enough it being determined by the presence of two-phase components with different types of lattices and, accordingly, different diffusion rate of the alloying elements, that are present in them. With an increase in the quenching temperature there happens not only correlation in the amounts of austenite and ferrite but also an alternation in the phases: ferrite is enriched with chromium, as compared to austenite. The chromium content in austenite at that can be in the area, where according to diagrams of Potak-Sagalevich, or Scheffler’s diagrams martensite transformation at cooling is possible. The highest austenite stability in chromium-nickel austenite-ferrite steels is ensured at the average chromium content $> 21$ wt.%.

The analysis of changes in microhardness of the phase components, depending on chromium content and under its influence shows the following (see Table 1).

| Steel grade   | Hardness, HRB | Phases content, % | Microhardness, HV |
|---------------|---------------|-------------------|-------------------|
| 10Cr15Mn10    | 95            | 57                | 43                | 277                | 331                |
| 09Cr17Mn9     | 95            | 47                | 52                | 279                | 386                |
| 09Cr18Mn10    | 96            | 21                | 79                | 244                | 335                |
| 09Cr20Mn10    | 94.5          | 61                | 38                | 277                | 316                |
| 09Cr21Mn10    | 94            | 31                | 69                | 241                | 376                |
| 12Cr18Ni10Ti  | 89            | 0                 | 100               | -                  | 277                |

At relatively smaller amount of ferrite 21-31% in the steels structure (09Cr18Mn10 and 09Cr21Mn10 steels) its microhardness has the smallest values 241-244 HV (see Table 1). As the amount of ferrite is increased up to 57-61% (10Cr15Mn10 and 09Cr20Mn10 steels) its microhardness grows up to 277 HV (Table 1). It can be explained by the solid solution strengthening of ferrite, evidently under the influence of its greater content of chromium and nickel. A similar picture is observed when austenite microhardness is changed, due to the changes in phase correlation under the influence of alloying. At the smallest austenite content ($A = 38 \%$) in 09Cr20Mn10 steel its microhardness is 316 HV, with an increase of its content to 69-78% its microhardness grows up to 335-376 HV (see Table 1). This can be explained by a combined stabilizing action of minor additions of copper and nickel.

It can be seen in Table 1 that austenite microhardness is higher than ferrite, it can be explained by a greater opportunity of carbon solving (chiefly), as well as austenite forming alloying elements in the face-centered cubic lattice, it coordinating quite well with the data from literature. In order to analyze the data obtained graphs of dependence of mechanical properties upon chromium content (within 15-21 wt.% Cr) were compiled at different fixed and close manganese contents).

3.2 Effect of chromium and manganese content upon the mechanical properties of the developed and investigated corrosion-resistant Fe-Cr-Mn steels

Chromium content was varied within 15.0 % - 21.4 wt.% limits with the objective of altering the phase correlation between austenite and ferrite, as it known that chromium is a strong ferrite-forming element and also and also for optimization of the chemical composition for improving the mechanical properties
and corrosion resistance of the developed and investigated Fe-Cr-Mn steels. Figure 2, a represents alternations in the mechanical properties under the influence of chromium within 15.0–21.4 wt.% at a constant (within a narrow interval) alloying with the smallest in the investigated range amount of manganese (8-9 wt.%).

![Figure 2](image_url)

**Figure 2.** Effect of chromium at manganese content 8-9 wt.% (a) and 9-10 wt.% (b) upon the mechanical properties of the investigated Fe-Cr-Mn steels: 1 – ultimate tensile strength (UTS); 2 – yield strength (YS); 3 – hardness, HB; 4 - percentage elongation (PE); 5 – Z; 6 – KCU.

The strength properties are changed with the presence of maximum (UTS=789 MPa, YS=589 MPa) at chromium content in steel 17 wt.%, while the percentage elongation is altered within a narrow interval 40-51 %. Still, there is a tendency towards reduction in percentage elongation (PE) from 51 % to 37-41 % with an increase of chromium in steels. At bigger alloying of steels with manganese within 9-10 wt.% limit, with an increase of chromium content the strength properties are changed in accordance with the curved lines with maximums UTS=830 MPa; YS=620 MPa, corresponding to 18.55 wt.% Cr (Figure 2, b). Hardness changes correlate with changes in strength.

Percentage reduction of area (Z) is also changed in the shape of a curved line with (Z=50%) maximum, however. It is observed in steels at 17 wt.% Cr content (see Figure 2, b). Percentage elongation has the highest values (PE=30%) at 17 wt.% Cr content and (PE=49%) at 20.7 wt.% Cr. Impact strength (KCU) is decreased from 294 J/cm² (2.94 MJ/m²) to 220 J/cm² (2.2 MJ/m²) with the increase of chromium content. At the highest alloying of steels with manganese (within the investigated concentration range) 10-11 wt.% when the chromium content is raised the strength properties are changed likewise drastically, still, the maximum (UTS=790 MPa; YS=610 MPa) is shifted towards lower chromium content 16.79 wt.% (Figure 3).
Figure 3. Chromium influence at manganese content 10-11 wt.% upon the mechanical properties of the investigated Fe-Cr-Mn steels: 1 – UTS, 2 – YS, 3 – HB, 4 – PE, 5 – Z, 6 – KCU.

At that the highest values of plastic characteristics: PE=64 % and Z=56 % correspond to chromium content 19.6 wt.%, while the impact strength KCU=320 J/cm² (3.2 MJ/m²) reaches its highest value when the content of chromium is 20.6 wt.%. However, for the highest strengthen indices quite high plasticity characteristics were reached: PE=39 %; Z~60 %; and for impact strength KCU=290 J/cm² (2.9 MJ/m²).

A comparison of the characteristics of mechanical properties of the developed nickel-free stainless steels on Fe-Cr-Mn basis with the well known throughout the world and widely applied chromium-nickel stainless steels of 18-8, 18-10 types, like (10-12)Cr18Ni9Ti (austenite class) and 08Cr21Ni6Mo2Ti (austenite-ferrite class) shows that the new steels possess greater strength properties (UTS, YS) at 180-240 MPa, the plasticity properties being similar or even higher (PE, Z), impact strength values being also similar or higher (KCU) (see Table 2). It is also quite worthwhile mentioning here that we used for our investigations and testing molded steels, while standard steels are manufactured by hot rolling. It is quite evident that the advantages of the new steels are bound to be higher after hot rolling or forging. For cast state of these new steels the reached advantages are quite high and are in good accordance with the requirements of the existing standards (GOST 7350-77 and AISI 304, ASTM A240).

It is important to point out here that passing of γ→α′ DIMTT plays a major part for formation of such lofty complex of mechanical properties of all developed and investigated nickel-free corrosion-resistant (stainless) steels. This transformation leads in the process of testing to the effects of additional self-strengthening and self-relaxation of stresses, thus drastically simultaneously increasing strength, plasticity and impact strength. The improved complex of properties of the designed cost-saving alloyed Fe-Cr-Mn steels can be explained primarily by that.
Table 2. Comparative characteristics mechanical properties (tensile, impact strength, ISO 6892-2015) of designed and standard corrosion-resistant steels after quenching at 1,050 (1050) °С.

| Steel grade | Ultimate Tensile Strength (UTS), MPa | Yield Strength (YS), MPa | Percentage elongation (PE), % | Percentage reduction of area (Z), % | Impact strength (KCU), MJ/m² |
|-------------|-------------------------------------|-------------------------|--------------------------------|-----------------------------------|-----------------------------|
| **Designed Fe-Cr-Mn (Ni-free) steels** | | | | | |
| 09Cr17Mn9  | 790 | 590 | 51 | 38 | 2.4 |
| 09Cr19Mn10 | 830 | 620 | 30 | 47 | 2.3 |
| 09Cr17Mn11 | 790 | 610 | 39 | 60 | 2.9 |
| 09Cr20Mn11 | 690 | 530 | 56 | 64 | 2.9 |
| **Standard Fe-Cr-Ni steels** | | | | | |
| 10Cr8Ni9Ti (GOST7350-77) | 530 | 230 | 38 | 55 | 2.5 |
| AISI 304 (ASTM A240) | 510 | 205 | 43 | - | - |
| 08Cr21Ni6Mo2Ti | 588 | 340 | 20 | - | 0.6 |

4 Conclusions
1. New cost-saving alloyed Fe-Cr-Mn (nickel-free) corrosion-resistant steels of austenitic-ferritic structural class were developed that possess an improved level of mechanical properties.
2. By varying the chromium content within 15 – 21.4 wt.% the manganese content within 8 – 11.4 wt.% in various combinations it was possible to achieve different phase relations between ferrite and austenite content, the governing influence belonging to chromium, while manganese exerting influence chiefly upon the degree of austenite metastability.
3. It was found out that by regulating the content of chromium and manganese in steels it was possible to control the relationship between the amounts of austenite and ferrite, as well as the degree of metastability of γ-phase. For replacement of costly nickel in steel it is advisable to add manganese, some little additions of copper and silicon and perform micro-alloying with strong carbide-forming elements, it allowing reaching of a complex of mechanical properties that can be classified as high enough.
4. Determined were optimal compositions of cost-saving alloyed Fe-Cr-Mn (nickel-free) corrosion-resistant steels (in chromium, manganese and carbon content with regard to additional alloying with small additions of copper, silicon and micro-alloying with carbide-forming elements) that ensure an improved complex of mechanical properties after quenching.
5. The improved complex of strength, plasticity properties and impact strength in the developed corrosion-resistant (stainless) Fe-Cr-Mn steels can be explained, on the one hand, by the number of austenite and ferrite phases and, on the other hand, by the kinetics and the volume of development of deformation martensite γ→α' transformation, occurring in the process of testing of mechanical properties.
6. Comparative testing has shown that the developed cost-saving alloyed (nickel-free) corrosion-resistant steels are superior to the existing costly and scarce chromium-nickel steels of 18-8 and 18-10 (10Cr8Ni9Ti) type of austenite class and 21-6 (08Cr21Ni6Mo2Ti) of austenitic-ferritic class, being a worthy alternative to them for practical applications.

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