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Original article
Оригинальная статья

Effect of silicon and vanadium on corrosion-mechanical properties of high-nitrogen Cr – Mn steels

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Abstract. The authors studied the phase composition, crystal lattice parameters, mechanical properties and stress corrosion resistance of high-nitrogen austenitic and austenitic-ferritic Cr–Mn steels after homogenizing treatment, aging and cold plastic deformation. It was established that alloying of Cr–Mn steels with silicon and vanadium can lead to the formation of different amounts of ferromagnetic δ-ferrite and, from its low content, to significant hardening due to the grain-boundary effect. The presence of δ-ferrite has a hardening effect both after homogenizing treatment and during cold plastic deformation. In vanadium-alloyed Cr–Mn steels, even after austenitization treatment at 1250 °C, a finer grain of austenite of 8 – 9 numbers is retained than those of steels alloyed with silicon, having after quenching from a lower temperature (1150 – 1170 °C) larger grain of 6 – 7 numbers. Formation of even small amounts of δ-ferrite leads to a decrease in corrosion cracking resistance of high-nitrogen chromium-manganese steels. At the same time, corrosion resistance of high-nitrogen steels with δ-ferrite is significantly lower than that of austenitic steels containing 0.4 % nitrogen and more single-phase Cr–Mn. Aging causes significant hardening of high-nitrogen, alloyed with both silicon and vanadium, Cr–Mn steels with δ-ferrite and is accompanied by a loss of ferromagnetism with a significant decrease in toughness and ductility. Disappearance of ferromagnetism seems to be due to the fact that δ-ferrite disintegrates into a σ-phase and a paramagnetic nitrogen-containing austenite. Microstructural and *X*-ray diffraction studies indicate that the aging of steel with δ-ferrite proceeds by a continuous mechanism, accompanied by a monotonous decrease in the lattice parameter of austenite due to the release of nitrides from it. Aging of two-phase steels, leading to the disappearance of δ-ferrite and ferromagnetism, caused a catastrophic decrease in corrosion cracking resistance.

Keywords: high-nitrogen Cr – Mn steels, δ-ferrite, microstructure, mechanical properties, stress corrosion resistance

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Влияние кремния и ванадия на коррозионно-механические свойства высокоазотистых Cr – Mn сталей

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Аннотация. Исследованы фазовый состав, параметры кристаллической решетки, механические свойства и коррозионная стойкость под напряжением высокоазотистых аустенитных и аустенито-ферритных Cr–Mn сталей после гомогенизирующей обработки, старения и холодной пластической деформации. Установлено, что легирование Cr–Mn сталей кремнием и ванадием может приводить к образованию разных количеств ферромагнитного δ-феррита и уже с малых его содержаний к существенному упрочнению, обусловленному зернограничным эффектом. Присутствие δ-феррита оказывает упрочняющий эффект как после гомогенизирующей обработки, так и при холодной пластической деформации. В легированных ванадием Cr–Mn сталях даже после аустенитизирующей обработки при 1250 °C сохраняется более мелкое зерно аустенита 8 – 9 номера, чем у сталей, легированных кремнием, имеющих после закалки от более низкой температуры (1150 – 1170 °C) большее по размеру зерно 6 – 7 балла. Образование даже небольших количеств δ-феррита приводит к снижению сопротивления коррозионному растрескиванию высокоазотистых хромомарганцевых сталей. При этом сопротивление коррозионному растрескиванию высокоазотистых сталей с δ-ферритом оказывается значительно ниже, чем у содержащих 0,4 % азота и более однофазных Cr–Mn аустенитных сталей. Старение вызывает существенное упрочнение высокоазотистых, легированных как кремнием, так и ванадием, Cr–Mn сталей с δ-ферритом и сопровождается потерей ферромагнетизма при значительном уменьшении ударной вязкости и пластичности. Исчезновение ферромагнетизма, по-видимому, обусловлено тем, что происходит распад δ-феррита на σ-фазу и парамагнитный азотсодержащий аустенит. Микроструктурные и рентгеноструктурные исследования свидетельствуют о том, что старение стали с δ-ферритом протекает по непрерывному механизму, сопровождающемуся монотонным снижением параметра решетки аустенита в связи с выделением из него нитридов. Старение двухфазных сталей, приводящее к исчезновению δ-феррита и ферромагнетизма, вызвало катастрофическое снижение стойкости против коррозионного растрескивания.

Ключевые слова: высокоазотистые Cr – Mn стали, δ-феррит, микроструктура, механические свойства, коррозионная стойкость под напряжением

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Introduction

When assessing the prospects, sequence of adoption, and results of carbon-free technologies in metallurgy, it has been noted [1] that nitrogen could serve as an alternative to carbon as a strengthening element in steel, providing greater strengthening than carbon in traditional steels [2; 3]. Corrosion-resistant high-nitrogen steels have been developed, containing at least 12 wt. % chromium, which exhibit high static and cyclic strength, wear resistance, and enhanced capability for plastic deformation while maintaining good ductility and impact toughness [4; 5]. It is known that austenitic Cr–Ni and Cr–Mn steels, as well as corrosion-resistant martensitic and austenitic-martensitic steels, have low resistance to stress corrosion cracking (SCC) [6]. Higher long-term corrosion resistance is observed in ferritic and austenitic-ferritic steels. Austenitic high-nitrogen Cr – Mn steels, which are also highly resistant to hydrogen embrittlement and possess high corrosion fatigue strength, have demonstrated resistance to SCC in various environments [7 – 10].

The studies [10; 11] examined the influence of silicon on the fine structure and wear resistance of high-nitrogen Cr–Mn steels under dry sliding friction. It was established that alloying with silicon improves resistance to adhesive wear while maintaining low friction coefficients (f = 0.25 – 0.33). The effect of silicon on the tribological properties of these steels is associated with the activation of planar dislocation slip. Additionally, in [10], it is noted that metastable austenitic Cr–Mn steels, alloyed with 0.15 – 0.25 wt. % nitrogen, exhibit increased resistance to cavitation-erosion damage [12] and abrasive wear [13], which is largely explained by the formation of deformation-induced α-martensite under contact loading. The high resistance of Nitronic 60 steel to adhesive wear is attributed by the authors [14; 15] to the low stacking fault energy of austenite, the steel's ability for intensive strain hardening, and the formation of oxide films on the friction surface that prevent galling.

It is of interest to evaluate the SCC resistance of nitrogen-containing Cr–Mn steels with an austenitic-ferritic structure. The aim of this study is to investigate the influence of certain ferrite-forming elements (such as silicon and vanadium) on the corrosion-mechanical properties of Cr–Mn steels.

Materials and methods

The steels were melted under normal conditions at atmospheric pressure in a 60-kg induction furnace. The chemical composition of the studied steels is shown in Table 1. The sulfur and phosphorus content in all the melted steels did not exceed 0.01 and 0.04 wt. %, respectively. The ingots of nitrogen-containing steels were homogenized at 1150 °C for 8 – 15 h and forged into bars with a cross-section of 20×20 mm^2. From these, standard fivefold tensile test specimens with a working diameter of 5 mm and standard specimens with a 10×10 mm^2 cross-section and a *U*-shaped notch were prepared for impact toughness testing.

Table 1. **Chemical composition of high-nitrogen Cr – Mn steels and content of δ-ferrite in them**

Таблица 1. **Химический состав высокоазотистых Cr – Mn сталей и содержание в них δ-феррита**

Steel grade	Element content, wt. %						Amount of δ-ferrite, %
	N	Si	V	Cr	Mn	C	
10Kh16G17S4A0.3	0.28	4.50	0.09	16.0	17.1	0.11	3
12Kh19G19S2A0.5	0.50	2.37	0.13	19.3	19.4	0.13	0
10Kh19G20S4A0.5	0.52	4.30	0.18	19.6	20.3	0.10	32
07Kh18G19FA0.4	0.42	0.49	1.04	17.5	18.9	0.07	5
07Kh19G18FA0.7	0.73	0.35	1.07	18.8	18.0	0.07	0

The ultimate tensile strength and yield strength of the steels were determined with an error margin of ±5 MPa, and the elongation was measured with an accuracy of 0.1 %. SCC (stress corrosion cracking) tests were conducted using a specially developed method [16] in a 20 % aqueous sodium chloride solution in distilled water at room temperature and at stresses ranging from 0.80 to 0.95 of the yield strength.

The microstructure was studied using an Axio Observer.D1m optical microscope. Magnetic measurements were performed on an α-phase meter, which records the content of δ-ferrite.

X-ray diffraction studies were conducted on a DRON-4-07 diffractometer using iron radiation. Qualitative and quantitative phase analysis was performed using the Rietveld method [17], following the optimization of interference maxima. The accuracy of the quantitative phase analysis was ±5 %. Precision measurements of the austenite lattice parameter were carried out using the final interference lines $311_{\alpha 1}$ and $222_{\alpha 1}$, recorded in discrete mode with a step of 0.02° [18; 19].

Results and discussion

X-ray diffraction and magnetometric phase analysis indicate that Cr – Mn steels 12Kh19G19S2A0.5 and 07Kh19G18FA0.7 are in the austenitic state (Table 1). In steels with lower nitrogen content, a small amount (3 – 5 %) of the ferromagnetic δ-ferrite phase is additionally present, while in steel with 4 % silicon, the δ-ferrite content reaches 32 %. Dispersed grains of δ-ferrite of various shapes and sizes are mainly located along the boundaries of austenite grains (Fig. 1).

Steels alloyed with silicon, after homogenizing treatment at 1150 – 1170 °C, exhibited the same austenite grain size, corresponding to 6 – 7 numbers. In vanadium-alloyed steels, even after austenitizing treatment at 1250 °C, a finer austenite grain size of 8 – 9 numbers was retained.

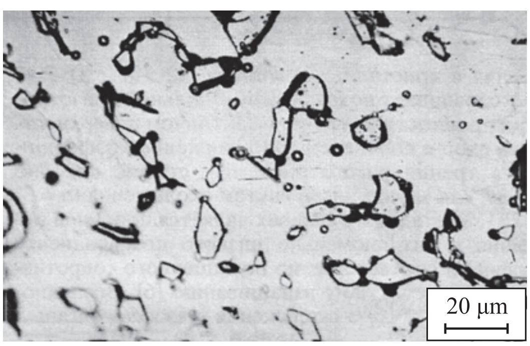

Fig. 1. Microstructure of quenched from 1100 °C 10Kh19G20S4A0.5 steel with δ-ferrite

Рис. 1. Микроструктура закаленной от 1100 °C стали 10Х19Г20С4А0,5 с δ-ферритом

The mechanical properties of the steels after homogenizing treatment are shown in Table 2. For comparison, the lower part of the table provides the mechanical properties of similar Cr – Mn austenitic steels with a similar nitrogen concentration but not alloyed with silicon or vanadium. It can be seen that at equal nitrogen concentrations, steels containing δ-ferrite have higher strength properties and lower values of elongation and impact toughness compared to austenitic steels not alloyed with silicon or vanadium, which promote the formation of ferrite.

High-nitrogen Cr – Mn steels containing δ-ferrite, like similar austenitic steels, are significantly hardened during cold plastic deformation while maintaining good ductility. The hardening degree (Δσ/Δε) of steels with different nitrogen and silicon concentrations, as well as the critical degree of cold deformation (e_{cr}) required to achieve a yield strength of 1200 N/mm^2 in some critical components (e.g., retaining rings of powerful turbogenerators), are shown in Table 3.

In terms of these parameters, austenitic-ferritic steels containing 0.3 – 0.5 % nitrogen and 4 % silicon were found to be comparable to steels not alloyed with silicon but containing a higher nitrogen content. At the same time, alloying austenitic steel 12Kh19G19S2A0.5 with 2 % silicon had virtually no effect on the hardening degree or the critical deformation degree e_{cr}. These parameters were similar to those of steel 08Kh18G18A0.5. Thus, it can be inferred that the presence of δ-ferrite has a hardening effect (grain-boundary hardening), both after homogenizing treatment and during cold plastic deformation.

At the same time, even the formation of small amounts of δ-ferrite reduces the stress corrosion cracking (SCC) resistance of high-nitrogen Cr – Mn steels. For instance, while the formation of 3 % δ-ferrite in steel 10Kh16G17S4A0.3 did not significantly affect

Table 2. **Mechanical properties of high-nitrogen steels after homogenizing treatment**

Таблица 2. **Механические свойства высокоазотистых сталей после гомогенизирующей обработки**

Steel grade	$\sigma_{0.2}$, N/mm^2	σ_u, N/mm^2	δ, %	Ψ, %	*KCU*, J/cm^2
10Kh16G17S4A0.3	465	908	66	63	287
12Kh19G19S2A0.5	522	924	65	72	–
10Kh19G20S4A0.5	590	982	52	61	–
07Kh18G19FA0.4	530	890	46	63	122
07Kh19G18FA0.7	720	1100	43	62	181
05Kh14G20A0.3	350	720	68	74	–
08Kh18G18A0.5	530	910	67	73	360
08Kh19G19A0.7	570	990	63	72	300

Table 3. **Hardening degree (Δσ/Δε) during cold plastic deformation by 15, 30, 40 % and critical deformation degree (e_{cr}) for different Cr – Mn steels**

Таблица 3. **Степень упрочнения (Δσ/Δε) при холодной пластической деформации на 15, 30, 40 % и критическая степень деформации (e_{cr}) для разных Cr – Mn сталей**

Steel grade	Δσ/Δε for various degrees of deformation, %			e_{cr}, %	SCC test duration, h
	15	30	40		
10Kh16G17S4A0.3	56.0	36.2	30.5	37	450 – 500 ∨
12Kh19G19S2A0.5	56.0	37.0	31.2	36	3000 ∧
10Kh19G20S4A0.5	65.4	41.6	34.2	27	300 – 2000 ∨
05Kh14G20A0.3	42.0	29.0	24.7	50	430 – 550 ∨
08Kh18G18A0.5	55.0	36.7	31.0	36	5000 ∧
08Kh19G19A0.7	66.0	42.0	34.7	26	5300 ∧

N o t e : ∨ – samples failed during SCC testing; ∧ – samples were withdrawn from testing without signs of SCC.

SCC resistance compared to the similar silicon-free austenitic steel 05Kh14G20N4A0.3, which has low resistance, the presence of 32 % δ-ferrite in steel 10Kh19G20S4A0.5 drastically reduced its SCC resistance compared to the austenitic steel 08Kh18G18A0.5, which is not susceptible to SCC [20 – 22] (Table 3). In the case of silicon alloying while retaining the austenitic structure (steel 12Kh19G19S2A0.5), no reduction in SCC resistance is observed. Similarly, the presence of δ-ferrite affects vanadium-alloyed steels. For example, the two-phase steel 07Kh18G19FA0.4 is prone to SCC within 250 – 750 h under stresses of 1050 – 1150 N/mm^2, while austenitic steel 07Kh19G18FA0.7, subjected to the same stress levels, was removed from testing after 5000 h without any signs of SCC.

Aging causes significant hardening of high-nitrogen Cr – Mn steels alloyed with both silicon and vanadium, converting them into a non-magnetic state. For example, the yield strength of the vanadium-containing high-nitrogen steel 07Kh18G19FA0.4 with 5 % δ-ferrite increases by 290 N/mm^2 after 16 h at 650 °C, whereas in the austenitic steel 07Kh19G18FA0.7, it increases by only 190 N/mm^2 compared to homogenizing treatment, with a significant decrease in impact toughness and ductility in both steels. The negative effect of vanadium and silicon on ductility and impact toughness is also noted in aging carbon-containing austenitic steels [23]. It is worth noting that in the high-nitrogen steel alloyed with silicon and containing 32 % δ-ferrite, hardness increases to 35 – 37 HRC after 2 – 4 h at 700 °C, comparable to the hardness of highly tempered alloy steels with 0.4 % carbon.

It should be noted that after aging, steels with δ-ferrite become non-magnetic, apparently due to its decomposition (according to the phase diagram) into the σ-phase and paramagnetic nitrogen-containing austenite. In this case, the content of δ-ferrite decreases monotonically with increasing aging time (Fig. 2). Microstructural and *X*-ray diffraction studies (Fig. 3) indicate that the aging of steel with δ-ferrite proceeds by a continuous mechanism, accompanied by a gradual decrease in the austenite lattice parameter due to the precipitation of nitrides. At the same time, in high-nitrogen Cr – Mn austenitic steels containing more than 0.3 % nitrogen and not alloyed with silicon, the intermittent decomposition of austenite becomes significantly more pronounced during aging [24].

Aging of two-phase steels, leading to the disappearance of δ-ferrite, resulted in a catastrophic decrease in SCC resistance. Aged samples experienced stress corrosion cracking under the selected test conditions within 10 – 70 h.

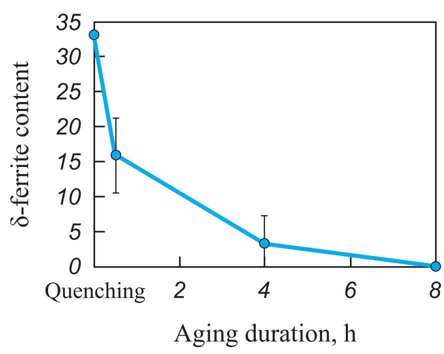

Fig. 2. Change in the content of δ-ferrite in 10Kh19G20S4A0.5 steel depending on duration of aging at 700 °C

Рис. 2. Изменение содержания δ-феррита в стали 10Х19Г20С4А0,5 в зависимости от продолжительности старения при 700 °С

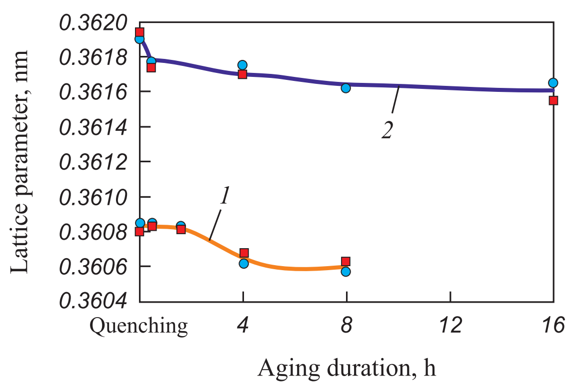

Fig. 3. Dependence of austenite lattice parameter of steels 10Kh16G17S4A0.3 (*1*) and 10Kh19G20S4A0.5 (*2*) on duration of aging at 700 °C:
● – calculation by line $311_{\alpha1}$; ■ – calculation by line $222_{\alpha1}$

Рис. 3. Зависимость параметра решетки аустенита сталей 10Х16Г17С4А0,3 (*1*) и 10Х19Г20С4А0,5 (*2*) от продолжительности старения при 700 °С:
● – расчет по линии $311_{\alpha1}$; ■ – расчет по линии $222_{\alpha1}$

Conclusions

Alloying high-nitrogen Cr – Mn steels with silicon or vanadium leads to the formation of δ-ferrite and significant hardening, both after homogenizing treatment and during cold plastic deformation, which is due to the grain-boundary effect.

In vanadium-alloyed Cr – Mn steels, even after austenitizing treatment at 1250 °C, a finer austenite grain size (8 – 9 numbers) is retained compared to silicon-alloyed steels, which exhibit a coarser grain size (6 – 7 numbers) after quenching at 1150 – 1170 °C.

The formation of even small amounts (3 – 5 %) of δ-ferrite in high-nitrogen Cr – Mn austenitic steels, along with hardening, leads to a reduction in stress corrosion cracking resistance.

Aging is accompanied by a further reduction in stress corrosion resistance, the disappearance of magnetism, and significant hardening of high-nitrogen Cr – Mn steels containing δ-ferrite, likely due to its decomposition into the σ-phase and nitrogen-containing paramagnetic austenite, as well as the precipitation of nitrides.

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