New approach to development and manufacturing technologies of duplex steel

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We conducted a brief review of current production and application of duplex and super duplex steels for manufacture of equipment exposed to the hazard of sulphide stress-corrosion cracking, sea water and other corrosive environment. The super duplex steel with enhanced corrosion-mechanical characteristics in comparison with the known steels of austenitic-ferritic class was developed. Based on the concepts of formation of a special structure of two-phase austenitic-ferritic steels in the process of crystallization, the possibilities of compositional, technological, thermal and special impact techniques are considered and advanced ways of controlling physical, chemical, structural homogeneity and properties of super duplex steels are developed. Electroslag remelting with the application of low-frequency alternating current provides effective control over the length of the two-phase area, the size of the primary dendrites of the austenitic and ferritic phases, the average distance between their axes, the parameters of the crystallizing cell, the development of liquation phenomena and the size of the growing non-metallic phases. Within framework of the proposed approach, the thermodynamic and kinetic conditions for the formation and growth of hardening phases are assessed, a new composition and a complex technology for the manufacture of corrosion-resistant super duplex steels for gas and oil production equipment has been developed. Thermodynamically stable, having sizes of 30-300 nm, niobium nitrides and carbonitrides are located inside the grains of the ferritic phase. At the same time, the sigma phase and chromium carbide precipitates at the intergrain boundaries are not observed. The results of the determination of mechanical and corrosion properties in accordance with the NACE TM 0177 standard (method A), tests of corrosion witness-samples in field conditions demonstrate the advantages and prospects of using new super duplex steel for the manufacture of oil and gas production equipment operating in an environment with high H₂S content and CO₂ under significant mechanical loads, without the risk of brittle fracture.

Keywords: *super duplex stainless steel (SDSS), electro slag remelting (ESR), low frequency current (LFC), niobium nitrides and carbonitrides, corrosion cracking resistance (CCR).*

1 Retrospective review of super duplex steels

The history of stainless nickel-chromium and nickel-chromium-molybdenum steels of austenitic class goes back more than 100 years, to the moment, when steels V2A and V4A were patented by Krupp. Two-phase austenitic-ferritic corrosion-resistant steel is a bit "younger". The Swedish company AVESTA Steelworks was the first to produce steel, which today is called duplex. Intensive use of duplex steels began along with the rapid development of the demands in the chemical industry in the 1980s, where the economic factors associated with nickel deficiency played a significant part. High strength and increased resistance to chloride and sulphide stress corrosion cracking compared to

traditional austenitic steels ensured the growing popularity of duplex steels.

The retrospective of duplex steels is represented in a number of works dated back to 1990s (Olsson J. [1], Charles J. [2], Gunn R.N. [3]) and of recent years (Alvares-Armas [4], Ciuffini A.F. [5], M. Knyazeva [6]).

The enhancement of the corrosion-mechanical characteristics and the achievement of structural stability were attributed by the authors [2] to the trend of alloying of contemporary SDSS with Cr, Mo and N (Fig.1).

The possibilities of reducing the content of Ni and Mo are widely reported in the said works and catalogues of manufacturers for obtaining a cost-effective alternative to the well-known grade S32205. Various options are developed for increasing the content of Cr, N, Cu and W in combination with relatively lower content of Ni and Mo in relation to the commonly used steel S32750 (Table 1).

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Table 1. Chemical composition of reference duplex steels proposed by the leading European manufacturers Sandvik, Outokumpu, Schmolz-Bickenbach.

Grade, Standard	Chemical composition, wt %												PREN, API 610
	C	Si	Mn	Cr	Ni	Mo	N	S	P	Cu	W	Other	
ZI 130 (03Cr24Ni6Mo3N) Technical conditions 14-1-3880-84	≤ 0.03	≤ 0.4	≤ 2.0	23.5 25.0	5.8 6.8	2.5 3.5	0.05 0.15	≤ 0.02	≤ 0.035	—	—	—	36
DMV18,5 UNS S31500	≤ 0.03	1.4 2.0	1.2 2.0	18.0 19.0	4.25 5.25	2.5 3.0	0.05 0.10	≤ 0.03	≤ 0.03	—	—	—	29
DMV22,5 UNS S31803	≤ 0.03	1.4 2.0	≤ 2.0	21.0 23.0	4.5 6.5	2.5 3.0	0.06 0.20	≤ 0.02	≤ 0.03	—	—	—	33
SAF2304 UNS S32304	≤ 0.03	≤ 1.0	≤ 2.5	21.5 24.5	3.0 5.5	—	0.05 0.20	≤ 0.04	≤ 0.04	—	—	—	25
SAF2205 UNS S32205	≤ 0.03	≤ 0.5	≤ 2.0	21.5 24.5	4.5 6.5	3.0 3.5	0.14 0.20	≤ 0.015	≤ 0.035	—	—	—	36
SAF2507 UNS S32750	≤ 0.03	≤ 0.5	≤ 1.2	24.0 26.0	6.0 8.0	3.0 5.0	0.24 0.32	≤ 0.015	≤ 0.035	—	—	—	42
DMV2574 UNS S32760	≤ 0.03	≤ 1.0	≤ 1.0	24.0 26.0	6.0 8.0	3.0 4.0	0.20 0.30	≤ 0.01	≤ 0.03	—	0.5 1.0	—	40
SAF2906 UNS S32906	≤ 0.03	≤ 1.0	≤ 1.0	28.0 30.0	5.0 7.0	1.8 2.5	0.40	≤ 0.01	≤ 0.03	—	—	—	42
Forta LDX 2101 EN 1.4162,UNS S32101	≤ 0.04	≤ 1.0	4.0 6.0	21.0 22.0	1.35 1.90	0.1 0.8	0.20 0.25	≤ 0.01	≤ 0.03	0.10 0.80	—	—	27
1.4507(URANUS52N+) EN10088-2,2005	≤ 0.03	≤ 0.7	≤ 2.0	24.0 26.0	6.0 8.0	3.0 4.0	0.20 0.30	≤ 0.015	≤ 0.035	1.0 2.5	—	—	41
A182 Grade F55	≤ 0.03	≤ 1.0	≤ 1.0	24.0 26.0	6.0 8.0	3.0 4.0	0.20 0.30	≤ 0.01	≤ 0.03	0.5 1.0	0.5 1.0	—	40
Zeron 100 1.4501	≤ 0.03	≤ 1.0	≤ 1.0	24.0 26.0	6.0 8.0	3.0 3.4	0.20 0.30	≤ 0.01	≤ 0.03	0.5 1.0	0.5 1.0	—	41
SAF2906 Safurex	≤ 0.02	0.30	1.0	29.0	6.6	2.0	0.35	≤ 0.01	≤ 0.03	-	-	-	41
SAF2707HD	≤ 0.03	≤ 0.5	≤ 1.5	26.0 29.0	5.5 9.5	4.0 5.0	0.30 0.50	≤ 0.01	≤ 0.035	-	-	Co 1.0	45
SAF3207 HD	≤ 0.03	≤ 0.5	≤ 1.5	29.0 33.0	6.0 9.0	3.0 5.0	0.40 0.60	≤ 0.01	≤ 0.035	-	-	-	50
X03Cr23Ni6Mo4Cu3NbN (after ESR) - new development by CNIITMASH	≤ 0.04	0.3 0.5	0.9 1.2	22.5 24.0	5.8 7.0	3.5 4.8	0.16 0.25	≤ 0.004	≤ 0.010	3.0 3.3	—	Nb 0.27 0.37	max46

Fig. 1. Structural stability of duplex steels taking into account the data [2] and [7].

These developments are aimed at enhancement of strength characteristics and corrosion resistance of steel.

For a formalized assessment the PREN pitting corrosion protection coefficient is used, which is calculated from the content of alloying elements (Table 2). Sandvik informs of the development of hyper duplex high-alloyed steels SAF2707 HD and SAF3207 HD, where PREN tends to 50. Along with the said upgrades of the duplex steel composition, there is a particular interest to steels not having a high PREN value, however, alloyed with less expensive elements, for example S32101. The developers of such modifications of duplex steel make a choice in favor of improved produce-ability of products.

Table 2. Calculation of pitting corrosion protection coefficient PREN according to different sources.

Source	PREN
Lorenz (1969)	%Cr + 3,3 %Mo
Truman (1978)	%Cr + 3,3 %Mo + 16 % N
Herbsleb (1982)	%Cr + 3,3 %Mo + 30 % N
Gysel (1987)	%Cr + 3,3 %Mo + 1 %N
Heimgartner (1988)	%Cr + 3,3 %Mo + 15 % N + 2 %Cu
Rondelli (1995)	%Cr + 3,3 %Mo + 30 % N - 1 %Mn
API Standard 610/ ISO 13709 (2003)	[%Cr - 14,5%C] + 3,3 %Mo + 2%Cu + 2%W + 16 %N

The fundamental features of *SDSS* ensuring the combination of high-performance mechanical and corrosion characteristics are the optimal ratio of the austenitic and ferritic phases close to 1:1 [8-12], high degree of dispersion and mixing [13]. However, the possibility of precipitation of chromium carbides and intermetallic phases, first of all, the sigma phase during slow cooling indicates the potential danger of brittle fracture. Manufacture of semi-finished metallurgical products of *SDSS* provides modern requirements for their quality characteristics at the account of use of vacuum-carbon deoxidation, secondary slag refining and plastic deformation in hot and cold state, precision heat treatment methods in the process flow.

However, the reserves for increasing the physical, chemical, structural homogeneity of the *SDSS* are not exhausted and research in this area is still relevant.

2 New approach to development of *SDSS* composition

Upgrade of *SDSS* composition is aimed at elimination of the effect of weakening of grain boundaries with large chromium carbides formed due to high chromium content, replacing them with small niobium nitrides and carbonitrides, dispersion strengthening ferritic grains.

At the same time it is advantageous to ensure a homogeneous (uniform) distribution of ferritic and austenitic grains in the primary structure of the ingot, as well as necessary conditions of the efficient thermal deformation stage. Hence, the following areas of *SDSS* improvement may be proposed:

- Optimization of the morphology and composition of the secondary phases, for example, by obtaining stable nanoscale components of the structure;

- Choice of a rational production scheme that makes it possible to you to manage both the crystallization process and the thermal deformation cycle to stabilize the structure and, accordingly, the strength, viscoplastic and corrosion characteristics of steel.

Developing *SDSS* composition we took into account the following factors determining in their entirety the system of alloying:

1. Copper, concentrating on the surfaces of grains and the inner surface of micropores, has an inhibitory effect on the reaction rate of hydrogen sulfide and water present in the fluid with carbon in steel. According to [14], copper having concentrations of about 3% has a positive effect on the corrosion resistance of austenitic-ferritic steels in hydrogen sulfide environment, most effectively slowing down the sulfide-forming reaction and preventing hydrogen sorption, which was confirmed by the data [15] on the degree of plastic properties change during cathode hydrogenation. Due to the formation of eutectics of copper and its sulfide, the solid solution is strengthened by the copper-containing phase. According to the authors [12,16] it causes positive changes in macro-and microstructure after aging.

2. Alloying of *SDSS* with molybdenum, strengthening solid solution, at the same time inhibits the formation of methane in the micropores [17,18] due to the reduction in the number of combined contacts of carbon and hydrogen with iron, and has a positive effect on the resistance of steel to hydrogen corrosion.

3. Steel alloying with nitrogen not only provides a partial replacement of nickel as an austenizing element, but, in combination with nitride-forming elements, first of all niobium, ensures high-performance strength and viscoplastic characteristics, while increasing the resistance of *SDSS* to pitting, sulfide, carbon dioxide and bacterial corrosion. Nitrides of alloying elements are not prone to coagulation in contrast to carbides, therefore they constrain the growth of austenitic grains. Thus, the strength increase is provided not only for grain boundary, but also for the dispersion hardening mechanism.

Corrosion resistance of steel is determined not only by its chemical composition and structure, but also by contamination with non-metallic inclusions that exhibit corrosion activity in specific operating conditions. The most sensitive places for pitting formation are sulfide inclusions, often having a substrate in the form of oxide. Mechanisms of influence of inclusions on the processes of pitting formation and development are associated with local deformation (increased stress level, characteristic mainly for "solid inclusions" of oxides), or activating action of sulfide ions, products of dissolution of "soft inclusions" of sulfides or oxysulfides in [19]. These factors cause the necessity to limit the content of sulfur and complex oxide inclusions, which create an increased level of stress in the surrounding matrix and thus contribute to the acceleration of corrosion processes.

The analysis of [12] the composition of nonmetallic inclusions exhibiting corrosion activity in aqueous media containing hydrogen sulfide, taking into account the pH potential of aluminates and calcium silicates, and complex sulfides (Ca, Mg, Mn)S, revealed the following sequence of dissolution of inclusions in descending order: $MgS \rightarrow CaS \rightarrow MnS \rightarrow mCaO \cdot nAl_2O_3 \rightarrow mCaO \cdot SiO_2 \rightarrow Al_2O_3 \rightarrow SiO_2$.

3 New approach to *SDSS* manufacturing process

Along with the traditional treatment of liquid steel in the ladle, an electroslag remelting (ESR) is used to reduce the total contamination of steel with non-metallic inclusions, remove almost completely point inclusions, reduce significantly the number of point sulfide and oxide inclusions and ensure their uniform distribution in the ingot. However, in addition to refining, the technology of electroslag remelting is able to provide chemical, physical and structural homogeneity of the ingot already in the process of crystallization and joint formation of ferrite and austenite grains. In contrast to the traditional ingot, characterized as a system resistant to physical and physical-chemical influences, ESR ingot actively responds to changes in the conditions of its

formation. Due to the intensive heat supply from the consumable electrode and its transfer to the crystallizer and the stood, the share of the latent heat of solidification varies widely in the overall heat balance of the ingot. It is possible to vary the volume (depth) and shape of the liquid metal bath within a sufficiently large range of values, combining the possibilities of thermal, technological and special methods having an effect on the crystallization process [20]. The length of the two-phase field and its thermophysical parameters, the effective impurities distribution coefficients and other parameters are varied in this case. Electroslag remelting, influencing the dynamics of the primary crystallization process, allows to control to a large extent the parameters of the primary crystal structure [21]. This effect is particularly noticeable when low-frequency alternating current (LFC) is used. According to [22] in this case, the size of the primary dendrites and the average distance between their axes are reduced, preventing the development of liquation phenomena and contributing to a decrease in the size of the growing non-metallic phases. Differences between them in different zones of the ingot are leveled, which reduces the profile of zonal liquation.

Simulation of the phase composition of *SDSS*, taking into account the thermodynamic properties of the components and their compounds, kinetic factors influencing the growth of particles of the carbides, nitrides and carbonitrides and determining their size [23], allowed to determine the temperature range for the formation of stable nitrides and carbonitrides of niobium on the inside of the ferritic phase in the course of alloying of steel Cr-Ni-Mo-Cu-Nb-N, it is 650-1000 °C (Fig. 2). Accelerated cooling of steel in the specified range provides fixing the average size of these particles in the range of 30-300 nm [24].

Fig. 2. *SDSS* phase composition calculation results in Thermo-Calc for Windows 6. The target direction of formation of the phase composition is indicated by an arrow, and the interval of heating for hardening by a background.

The forgings of *SDSS* X03Cr23Ni6Mo4Cu3NbN (after ESR) weighing up to 50 kg were manufactured at

the experimental production facilities of CNIITMASH [25]. Ingots was forged at the temperature no less than 1050 °C on a hydraulic hammer with upsetting factor is a 4 and heat treated at 1055-1070 °C with holding 2 hours and rapid cooling in water.

4 Quality research of forgings of new *SDSS*

In a two-phase steel microstructure (Fig. 3) the share of δ -ferrite is $\approx 57\%$ which was measured on volumetric ferritometer MF-510 due to GOST 2246-70. A ferritic grain size under fourfold forging reduction is evaluated according to GOST 5639-82 as number G8-11, and the average spacing between the grains of austenite is 20 μm , which meets the international requirements to equipment for offshore petroleum production [26].

The amount of intermetallic NbCr₂ ranging in size from 2 to 10 μm (Fig.4) does not exceed 0.5%.

Sigma phase and chromium carbides of Cr₂₃C₆ type are detected in the structure. Particles of niobium carbonitrides and nitrides have characteristic dimensions of 30 - 300 nm. They are located mainly in the ferrite matrix.

Fig. 3. Microstructure of X03Cr23Ni6Mo4Cu3NbN (after ESR) steel sample, photographing with microscope Axiovert 40MAT (Carl Zeiss), with digital image analyzer ProgRes C3 and computer digital system for optical metallography. The microstructure is detected by means of electrolytic pickling in 10% chromium anhydride.

Fig. 4. Photos of X03Cr23Ni6Mo4Cu3NbN (after ESR) steel samples received with the help of scanning electronic

microscopes JED-2300 and VEGA3: a) austenitic and ferritic phase (large bright particles - intermetallics NbCr₂); b)

nanoscale niobium nitrides in the ferritic matrix.

Table 3. Characteristics of steel X03Cr23Ni6Mo4Cu3NbN (after ESR) and reference SDSS

Steel grade	σ_B , MPa	$\sigma_{0.2}$, MPa	δ , %	ψ , %	KCV ²⁰ , J/cm ²	HRC, at most	Source
			not less				
EN1.4410/SAF2507/ UNS S32750	730-900	530-650	20	40	100	33	[27]
EN1.4462/SAF 2205/ UR 45N+/UNS S31803	≥620	≥448	25	—	100	26	
EN1.4477 /SAF 2906/ UNS S32906	≥750	≥550	25	—	120	30	
EN1.4362/SAF2304/ UNS S32304	630-820	≥400	25	—	—	30	
EN1.4658/SAF2707HD/ UNS S32707	≥ 920	≥700	30	—	230 ¹⁾	34	
EN1.4469/ EN10213 UNS S32101	≥740	≥550	22	50	56 ²⁾	—	[28]
EN 1.4507/UR 52N+	≥770	≥550	25	—	—	32	[29]
EN1.4501/ UNS S32760	800-1000	≥550	20	—	100	30	[30]
ZI 130 (03Cr24Ni6Mo3N)	≥690	≥390	25	40	60	32	[31]
EN 1.4162/UNS S32101 Forta LDX 2101	650-850	≥450	≥30	—	—	31	[32]
X03Cr23Ni6Mo4Cu3NbN (after ESR)	820-880	630-680	25	55	58-130	30	[33]

Notes: ¹⁾ pipes with wall thickness less than 4 mm; ²⁾ cast parts of pumps

The study of the distribution of elements in the characteristic x-radiation detected the enrichment of ferrite with chromium and molybdenum, and austenite with nickel and copper. In micropores there is an increased content of copper (Fig. 5).

Fig. 5. Microstructure of steel X03Cr23Ni6Mo4Cu3NbN (after ESR) in x-radiation of copper.

The developed steel demonstrated a high level of strength and plastic characteristics, superior to those of reference SDSS (Table 3), during testing in "IT-Service" LLC and "Gazprom-VNIIGAZ" LLC, which determines the prospects of its use under high mechanical loads. The tests in LLC "Gazprom VNIIGAZ" of shock samples with a sharp incision "V" showed average value of fracture work 27.3 J at a temperature of -46 °C meeting the requirements of GOST R 51365-2009 to equipment intended for operation in climatic category «U» (KV⁻⁴⁶ > 20 J). Corrosion tests of CCR steel for 30 days (720 h), performed in accordance with the method

of NACE TM 0177 standard (method A), showed no damage and cracks. The rate of general corrosion in the gas phase of the test environment with equilibrium partial pressures of hydrogen sulfide and carbon dioxide 1.5 MPa each at a temperature of 60 °C was 0.0013mm/year. With an increase to 50 times the pitting has not been detected.

"Gazprom dobycha Urengoy" LLC experimental field tests of corrosion witness-samples in real media of piping of well No. 2081 KGS No. 208 GPUprAO of Gas Condensate Field No. 22 did not reveal any pitting, and the general corrosion rate amounted to 0.0007 - 0.0015 mm/year.

5 Conclusion

Super duplex steels have the potential to enhance physical, chemical, structural homogeneity and performance properties.

A new approach to development of the composition and manufacturing technology of SDSS made it possible to offer steel X03Cr23Ni6Mo4Cu3NbN (after ESR) having a high-dispersity austenitic-ferritic structure free from sigma phase and precipitates of chromium carbides. In the new steel, additionally alloyed with niobium and copper, nanoscale niobium nitrides provide dispersion hardening of the ferritic matrix, and the copper-containing phase (2-10 μm) concentrated on the surfaces of grains and micropores not only has an inhibitory effect on the reaction rate of hydrogen sulfide and water with steel carbon, but also strengthens the solid solution.

The new technology of SDSS production, including electroslog remelting, provides directed crystallization,

reduction in the length of the two-phase field compared to the production of cast ingots, control of its thermal parameters, effective distribution coefficients of impurities, etc. Acting on the crystallization process, electroslag remelting provides synchronous formation of austenite and ferrite grains, the growth of which is mutually limited due to a decrease in the average distance between their axes. Suppression of liquation phenomena helps to reduce the size of non-metallic phases.

The test results of "CNIITMASH" JSC, LLC "Gazprom VNIIGAZ", LLC "IT-Service", LLC "Gazprom dobycha Urengoy" and foreign experience of operation of duplex steels makes it possible to recommend the developed steel for the manufacture of equipment operating in environments containing H₂S and CO₂ in sea water under high mechanical loads, with a reduced risk of brittle fracture.

"CNIITMASH" JSC is ready to render technological assistance to machine-building plants in implementation of the manufacture of the equipment of new steel for oil and gas production, recycling of carbamide, sulphate pulping, desalination plants and seawater pipelines of nuclear power plants, product storage tanks, pressure vessels, etc.

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