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Influence of alloying with titanium and molybdenum on grain size high strength pipe steel

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Abstract. In Kazakhstan, there is a growing demand for oil and gas pipes and general-purpose pipes made of steels with high performance properties, high-quality blanks for mechanical engineering and structural steel for construction. The work was carried out within the framework of the Targeted Financing Program, in which, as one of the most urgent, the task was set to expand and improve the range of steel products of steel-smelting enterprises, which are in demand, first of all, in the domestic market. Some questions on microalloying with molybdenum and titanium are considered. The article presents the main results of experimental heats to obtain high-strength structural steel with titanium and molybdenum. Metallographic studies of experimental laboratory samples were carried out using an Olympus 51BX (TRF) light microscope. The metallographic analysis of the studied samples showed that the base steel 09G2S of the current production has a ferrite-pearlite structure. The grains have an acicular structure (the formation of polygonal ferrite) and correspond to 5-6 points. At the same time, heterogeneity is observed. The results of the study of laboratory samples alloyed with molybdenum and titanium (sample No.1 and No.2) confirm the effectiveness of the impact of microalloying elements on grain refinement, which is due to the release of a significant amount of carbonitrides of the type (MoCN, TiCN) along the grain boundaries. The microstructure of both samples consists of two well-defined phases - ferrite and pearlite, and the grain size corresponds to 7-8 points.

Keywords: *alloying, high-strength steel, precipitation hardening, carbonitride phases, grain refinement, ferrite, perlite*.

1. Introduction

The Republic of Kazakhstan occupies one of the leading places in terms of reserves of natural resources, including the ninth in terms of oil and gas. To date, daily oil and gas production is 195.9 thousand tons and 105.9 million m^3, respectively. Transportation of petroleum products to consumers is carried out under high pressure through welded steel pipes of large diameter. Severe climatic conditions for the operation of pipelines, aggressive elements such as sulfur, hydrogen and their compounds in transported products require the use of steels with high strength characteristics for the construction of oil and gas pipelines and limiting the content of harmful impurities (sulfur and phosphorus), gases (nitrogen and hydrogen) [1].

At present, the production of large-diameter spiral pipes in Kazakhstan is carried out from imported rolled products.

Mastering the production of steel for oil and gas pipelines at existing metallurgical plants located on the territory of the Republic of Kazakhstan will expand the range of products manufactured in the country and solve the problem of import substitution. Constantly growing requirements for increasing the productivity of main pipelines put forward the task of improving structural steels for large-diameter electric-welded pipes, fittings and valves.

To increase the productivity of pipelines, it is necessary to increase pressure and, consequently, use pipe steels of a higher strength class in order to avoid an increase in metal consumption (pipe wall thickness) and while maintaining or even increasing the toughness of steel, since an increase in pressure will inevitably lead to the risk of increased stresses in the pipe wall, capable of causing destruction of the pipeline [2].

The improvement of pipe steels, aimed at increasing the strength and toughness margin, is achieved both by changing the chemical composition (reducing the carbon content, modifying, microalloying and alloying), and by using modern metallurgical technologies (smelting, rolling and heat treatment), which provide an increase in quality of the metal (decrease in the content of harmful impurities, gases, nonmetallic inclusions, refinement of the structure, regulation of the phase composition) [3].

One of the well-known ways to improve the strength characteristics of pipe steels is alloying with alloys containing titanium, molybdenum, niobium and vanadium.

The main mechanism of steel hardening is grain refinement with dispersion hardening, controlled by precipitation of excess phases of carbonitrides of various types and fineness. The formation of carbonitride phases occurs even in a liquid solution through the chemical interaction of nitrogen and carbon atoms in steel with alloying elements introduced into it - titanium, molybdenum, niobium and vanadium [4].

The result of these reactions are solid and refractory microparticles of carbonitrides, which, with the achievement of the temperature of the onset of solidification of steel, play the role of centers for its bulk crystallization. It should be noted that many works are devoted to studying the behavior of carbonitride phases in high-strength low-alloy steels alloyed with strong carbonitride-forming elements, but they, for the

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most part, consider the processes and parameters of the precipitation of these phases at the final stages - during rolling and cooling [5].

Hardening by dispersed particles is very effective and has become quite widespread. Moving dislocations interact with dispersed particles and experience resistance to their movement by the mechanism described by Orovan (particle bending and creation of dislocation loops) or by other mechanisms (Hirsch, Ansel and Lenel, etc.) Hardening of the alloy the more dispersed particles, the smaller the distance between them and the greater the elastic modulus of the particles of the second phase, i.e. the stronger the particles themselves. It can be assumed that the most intense hardening is observed at the stage of formation of Cottrell clouds, when the coherent bond with the matrix is still preserved. Such separation can occur both during cooling after rolling or normalization, and during tempering [6].

Less attention has been paid to the study of the formation of carbonitride phases in liquid steel and the effect of already formed carbonitrides on primary crystallization. At the same time, it is well known that the grain size of steel, formed during the casting and crystallization of ingots and slabs, has a significant and inherited effect on their final structure, and hence on the mechanical properties of finished rolled products [4].

These requirements created the prerequisites for the development of steels with higher strength, increased impact strength and resistance to ductile and brittle fracture at construction and operation temperatures, as well as with good ductility, weldability in the "field" and factory conditions, with corrosion resistance. S_{tu} and cold resistance with a limited number of alloying elements.

Therefore, there is a need to create pipes of a higher strength category X100 (strength class K80) (σ_v 760-990 N/mm^2, σ_t 690-840 N/mm^2).

New requirements for sheet rolled pipe steels, the need to obtain high values of strength properties and at the same time achieve a high level of toughness require the search for new technological solutions by improving the technology of metallurgical processing in terms of optimizing the chemical composition (charge, alloying and microalloying elements) and developing thermomechanical rolling, including the use of accelerated cooling [7-9].

So, at the present time at the Chemical and Metallurgical Institute named after Zh. Abishev, work is underway to develop a technology for producing high-strength steel of X100 strength class.

Steel grade X100 is characterized by high strength and toughness combined with excellent weldability. These properties make it an ideal material for use in the offshore segment for oil and gas transportation - where pipes are constantly exposed to harsh environmental conditions such as waves, water currents and low temperatures.

The development of steel of X100 strength class is based on the concept of production of X80 steel with a high content of molybdenum, nickel, titanium, copper, vanadium and niobium, i.e. elements that increase impact strength, ductility, hardenability and weldability, and another mode of accelerated cooling. In the transition from X80 steel to X100 steel, the ferritic-bainite structure changes almost completely to bainitic.

In recent years, extensive material has been accumulated on the intermediate transformation of supercooled austenite and the properties of bainite in structural steels. The bainite structure has a complex nature, and depending on the carbon content, alloying elements and cooling conditions, it can significantly change its morphology. According to the formation temperature, upper and lower bainite are morphologically distinguished.

Features of the bainite transformation are associated with the occurrence at temperatures where there is no diffusion of iron atoms and alloying elements, but intense diffusion of carbon occurs. This causes, firstly, the presence of a wide variety of morphological forms of the resulting phases, and secondly, obtaining a different chemical composition of these phases, which differ in carbon content. The structure and properties of steel after transformation in the bainite region largely depend on the value of carbon redistribution [10-12].

An optimal structure with a bainitic α-phase can be obtained only with the correct choice of cooling conditions in the temperature range of phase transformations. In scientific papers [13-15] it is shown that the use of accelerated cooling provides an increase in the strength properties for steels of simple composition by 40- 50 N/mm^2, and for alloyed steels by 80-100 N/mm^2, in addition, it allows eliminate the banding of the structure and reduce the anisotropy of properties.

Summarizing the literature data [7-9, 11], accelerated cooling together with microalloying makes it possible to control the processes of structure formation of low-alloy steels by using additives of elements that increase the stability of austenite during cooling, as well as changing the rate of accelerated cooling and the temperature of completion of accelerated cooling various structural components and their combination in rolling can be obtained: ferrite of various morphology, ferrite-bainite or homogeneous bainite, as well as bainite-martensite microstructure.

The microstructure consisting of granular bainite as the matrix phase is the best choice for pipeline high strength steel. The structure of bainite in high-strength steels is formed due to alloying with elements that inhibit ferrite transformation, such elements include additions of Mn, Mo, Ni, Cr, Cu and Nb, which increases the volume fraction of bainite. Molybdenum forms carbides in steels, as soon as the carbon content in the steel becomes high enough, it is able to provide additional thermal hardening during tempering of hardened steels. It increases the creep resistance of low alloy steels at high temperatures. Molybdenum additives contribute to the refinement of steel grains, increase the hardening of steels by heat treatment, and increase fatigue strength.

Molybdenum predominantly enters the solid solution, distorting the lattice of the main solid solution, thereby strengthening it [6, 7].

Low-alloy hot-rolled steel alloyed with titanium (≈ 0.1%) is characterized by high strength, but low ductility and toughness, while the low content of titanium in steel (0.01-0.03%) refines its primary structure, since refractory titanium nitrides formed in liquid steel serve as crystallization centers.

The degree of recrystallization of such steel during hot rolling is lower than that of coarse-grained steel, therefore, its recrystallized austenite grain is more uniform and finer.

The binding of free nitrogen to titanium nitrides is the viscosity of the finished rolled product. After crystallization, titanium, in its excess with respect to nitrogen, binds sulfur and weakens the stringiness of manganese sulfides, forming sulfides and carbosulfides, which is accompanied by some improvement in plastic and viscous properties in the transverse direction [7].

Niobium forms NbC carbide with carbon, and NbN nitride with nitrogen, fine nitrides and carbonitrides of niobium are located along the boundaries of grains and subgrains, inhibit the movement of dislocations and thereby strengthen the steel. Niobium is an effective element for grinding grains of austenite and ferrite during heating for rolling or heat treatment. In addition, the effect of microalloying steel with niobium is to slow down the recrystallization of austenite during thermomechanical rolling due to dispersion strengthening and solid-solution transformation. The amount of niobium that passes into a solid solution when heated to a given temperature depends on the carbon content. A decrease in the carbon content in steel provides an increase in the solubility of niobium in austenite [16, 17].

Compliance with the correct ratio of alloying additives (Mo, Ti, Nb) helps to control weldability and provide an optimal balance of the content of carbon-nitride-forming elements that have a similar effect on the physical and mechanical properties of steel.

Based on the analysis of literary sources [4-7], it was established that the formation of a bainitic structure of optimal morphology is based on the following aspects of metallurgical science:

- reduction of carbon content (0.04-0.06%), which is favorable for toughness, weldability and segregation uniformity of the metal;
- increasing the degree of purity for harmful impurities and gases;
- complex microalloying of Mo, Ti and Nb in a stoichiometric ratio with nitrogen $Ti \geq 3.4N$;
- reduction of harmful impurities and gases ($\leq 0.002\%$ [S]; $\leq 0.010\%$ [P]; $\leq 0.006\%$ [N]; ≤ 2 cm3/100 g $[H_2]$);
- the use of thermomechanical rolling with accelerated cooling for maximum grain refinement.

In addition to the factors listed above, progress in the development of steels is accompanied by the improvement of metallurgical technology at all stages, including the desulfurization of cast iron, a complex of out-of-furnace processing, which ensures a high degree of purity of the metal.

2. Materials and methods

Experimental part. The employees of the laboratory of metallurgy and materials science of the Chemical and Metallurgical Institute named after. Zh. Abisheva conducted experimental melting to obtain high-strength structural steel with titanium and molybdenum. Changes in the microstructure of steel during its alloying with titanium and molybdenum have been studied.

For the base steel, as a comparison, we have chosen the 09G2S steel grade, which is close in its chemical composition to pipe steel grades, and which is mastered at the existing steel-smelting plants of the Republic of Kazakhstan.

Experimental melting to obtain high-strength steel with titanium and molybdenum was carried out in a Tamman high-temperature furnace, in alundum crucibles in an inert atmosphere. As a charge for smelting high-strength steel alloyed with molybdenum and titanium, metal samples were used, taken at the outlet from the converter of ArcelorMittal Temirtau JSC (AMT) of the following chemical composition: C - 0.03%; Mn - 0.04%; S - 0.04%, P - 0.06%.

After melting the scrap metal, upon reaching the required melt temperature of 1635°C, the steel was deoxidized with ferrosilicon (FeSi65) (GOST 1415-93), metallic manganese (Mn 998) (GOST 6008-90) and aluminum wire (99% Al) (GOST 295-79).

Alloying of steel was carried out using ferromolybdenum (FeMo60) (GOST 4759-91) and FeTi30 (GOST 4761-91) in the amount, respectively, %: FeMn-2.23; FeSi -0.47; A99.9-0.1; FeMo60-0.57 and FeTi30 - 0.18 and from the total mass of the metal charge. The consumption of deoxidizers and alloying materials is presented in Table 1.

Table 1. Consumption of deoxidizers and alloying agents

Mn998, g/ 100g	FS65, g/ 100g	Aluminum wire (99.0%) g/ 100g	FeTi30 g/ 100g	FeMo60, g/ 100g
2.23	0.47	0.1	0.18	0.57

The mass of ferroalloys to be added is calculated by the formula:

$$A=(B-C)\cdot D\cdot 100/(I\cdot F) \tag{1}$$

where: A – mass of ferroalloy, ton; B is the average content of the element in the finished steel, %; C is the content of the element in steel before deoxidation, %; D – mass of metal, considering the amount of metal from the previous heat, ton; I – is the content of the deoxidizing element in ferroalloys, %; F – assimilation of the deoxidizing element, %.

The deoxidation of the metal was carried out at the time of production based on obtaining the mass fractions of manganese, silicon and aluminum below their average values by 10.0 to 13.0%.

Table 2 shows the degrees of assimilation of elements during deoxidation and alloying.

Table 2. Assimilation of elements during deoxidation and alloying

Element	Assimilation, %
Silicon	95.00
Manganese	95.00
Aluminum	40.00
Molybdenum	85.0
Niobium	95.0

As a result of deoxidation and alloying with the indicated ferroalloys, prototypes of steels of the following chemical composition were obtained, table 3.

Table 3. Chemical composition of experimental steels

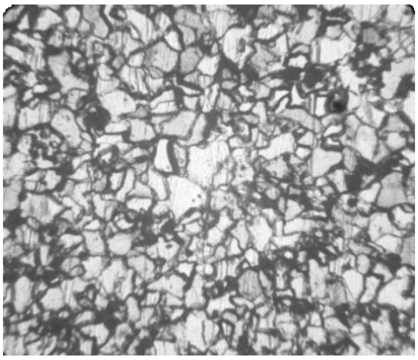
Steel grade	Content of elements, % mass							
	C	Si	Mn	P	S	Mo	Ti	Al
				not more than				
09G2S (basic)	0.09	0.6	1.5	0.021	0.012	-	0.02	0.04
Sample 1	0.05	0.15	1.6	0.011	0.005	0.21	-	0.03
Sample 2	0.06	0.16	1.5	0.013	0.004	-	0.03	0.04

To create the same heat treatment conditions, metal samples were placed together in one muffle furnace and heated to

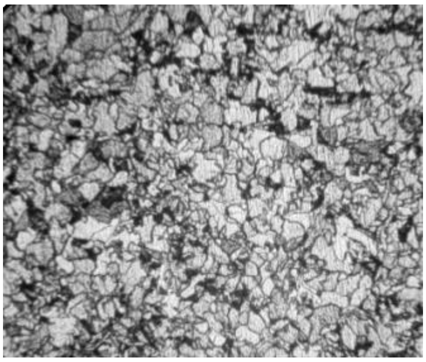
1100°C. Exposure of the samples in the oven at the specified temperature was 15 minutes. The samples were cooled with an oven to room temperature for 140 minutes.

Metallographic studies were carried out on an Olympus 51BX (TRF) metallographic microscope. The structure of the steel was determined according to GOST5638-82 (Methods for detecting and determining grain size) after pickling the samples in a 4% nitric acid solution.

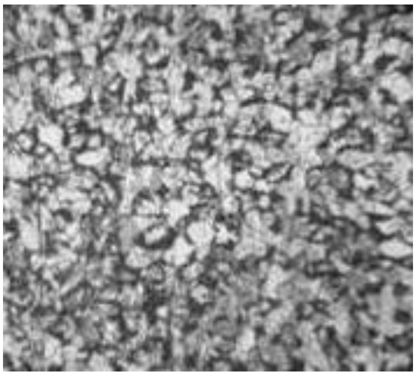
Figure 1 shows the microstructure of base steel 09G2S and experimental steels alloyed with molybdenum and titanium.



a) Grain score corresponds to 5-6. Steel 09G2S: 1 – ferrite (bright area); 2 – perlite (dark area)



b) Grain score corresponds to 7-8. Experimental steel (sample 1) with molybdenum: 1 – ferrite (bright area); 2 – perlite (dark area)



c) Grain score corresponds to 7-8. Experimental steel (sample 2) with titanium: 1 – ferrite (bright area); 2 – perlite (dark area)

Figure 1. Microstructure of comparative steel 09G2S and experimental steels - sample 1; sample 2, ×100

3. Results and discussion

Metallographic analysis of the studied samples showed that the base steel 09G2S has a ferrite-pearlite structure. Grain score corresponds to 5-6. Insufficiency is observed throughout the thin section.

The microstructure of steel alloyed with molybdenum (sample No.1) consists of fine grains of ferrite and pearlite, the grain score corresponds to 7-8. The grain structure of this steel is equiaxed.

The microstructure of steel alloyed with titanium (sample No. 2) consists of fine grains of ferrite and pearlite, the grain score corresponds to 7-8. The grain structure of this steel is equiaxed.

The results of a metallographic study of laboratory samples confirm the effectiveness of the impact of microalloying elements on grain refinement, which is due to the release of a significant amount of carbonitrides of the type (MoCN, TiCN) along the grain boundaries.

4. Conclusions

Metallographic analysis of the studied samples showed that the base steel 09G2S of the current production has a ferrite-pearlite structure. The grains have an acicular structure (the formation of polygonal ferrite) and correspond to 5-6 points. At the same time, heterogeneity is observed. The results of the study of laboratory samples alloyed with molybdenum and titanium (sample No.1 and No.2) confirm the effectiveness of the impact of microalloying elements on grain refinement, which is due to the release of a significant amount of carbonitrides of the type (MoCN, TiCN) along the grain boundaries. The microstructure of both samples consists of two well-defined phases - ferrite and pearlite, the grain size corresponds to 7-8 points.

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Беріктігі жоғары құбыр болатының түйіршік мөлшеріне титан және молибденмен легірлеудің әсері

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Аңдатпа. Қазақстанда мұнай-газ құбырларына және жоғары өнімділік қасиеттері бар болаттан жасалған жалпы мақсаттағы құбырларға, машина жасау үшін жоғары сапалы дайындамаларға және құрылысқа арналған құрылымдық болатқа сұраныс өсуде. Жұмыс нысаналы қаржыландыру бағдарламасы аясында жүргізілді, онда ең өзекті мәселелердің бірі ретінде болат балқыту кәсіпорындарының сұранысқа ие болат өнімдерінің номенклатурасын кеңейту және жетілдіру міндеті қойылды, ең алдымен, ішкі нарықта. Молибден және титанмен микроқорытпалау бойынша кейбір сұрақтар қарастырылды. Мақалада титан және молибден қосылған жоғары берік құрылымдық болат алу үшін тәжірибелік қыздырудың негізгі нәтижелері берілген. Тәжірибелік зертханалық үлгілердің металлографиялық зерттеулері Olympus 51BX (TRF) жарық микроскопының көмегімен жүргізілді. Зерттелетін үлгілердің металлографиялық талдауы қазіргі өндірістің 09G2S негізгі болатының феррит-перлит құрылымы бар екенін көрсетті. Дәндер иық тәрізді құрылымға ие (көпбұрышты ферриттің түзілуі) және 5-6 баллға сәйкес келеді. Сонымен бірге біркелкі еместігі байқалады. Молибденмен және титанмен легирленген зертханалық үлгілерді зерттеу нәтижелері (№1 және №2 үлгі) микроқорытпа элементтердің дәнді тазартуға әсер етуінің тиімділігін растайды, бұл дәнді дақылдың карбонитридтерінің едәуір мөлшерін бөлумен байланысты. түрі (MoCN, TiCN) астық шекаралары бойынша. Екі үлгінің де микроқұрылымы екі нақты анықталған фазадан – феррит пен перлиттен тұрады, дән мөлшері 7-8 баллға сәйкес келеді.

Негізгі сөздер: *легирлеу, жоғары берікті болат, дисперсиялық беріктендіру, карбонитридті фазалар, түйіршікті ұнтақтау, феррит, перлит.*

Влияние легирования титаном и молибденом на величину зерна высокопрочной трубной стали

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Аннотация. В Казахстане растет потребность в нефтегазовых трубах и трубах общего назначения из сталей, обладающих высокими эксплуатационными свойствами, заготовках высокого качества для машиностроения и конструкционной стали для строительства. Работы были выполнены в рамках Программы целевого финансирования, в которой, как одна из актуальных, была поставлена задача по расширению и улучшению сортамента стальной продукции сталеплавильных предприятий, востребованной, в первую очередь, на внутреннем рынке. Рассмотрены некоторые вопросы по микролегированию молибденом и титаном. В статье приводятся основные результаты опытных плавок по получению высокопрочной конструкционной стали с титаном и молибденом. Проведены металлографические исследования опытных лабораторных образцов в световом микроскопе Olympus 51BX (TRF). Металлографический анализ исследованных образцов показал, что базовая сталь 09Г2С текущего производства имеет феррито-перлитную структуру. Зерна имеют игольчатое строение (образование полигонального феррита) и соответствуют 5-6 баллам. При этом наблюдается, разнозернистость. Результаты исследования лабораторных образцов, легированных молибденом и титаном (проба №1 и №2) подтверждают эффективность воздействия микролегирующих элементов на измельчение зерна, что обусловлено выделением по границам зерен значительного количества карбонитридов типа (MoCN, TiCN). Микроструктура обоих образцов состоит из двух хорошо выраженных фаз - феррита и перлита, размер зерна соответствует 7-8 баллам.

Ключевые слова: *легирование, высокопрочная сталь, дисперсионное упрочнение, карбонитридные фазы, измельчение зерна, феррит, перлит.*