UDC 621.771.23-413.04

DOI: http://dx.doi.org/10.17580/cisisr.2017.01.06

# STUDY OF MICROSTRUCTURE OF ROLLED HEAVY PLATES MADE OF LOW-ALLOYED PIPE STEEL WITH INCREASED STRENGTH AND COLD RESISTANCE*

M. V. Chukin[1], P. P. Poletskov[1], O. A. Nikitenko[1], D. G. Nabatchikov[1]

[1] *Nosov Magnitogorsk State Technical University (Magnitogorsk, Russia)*

***E-mail: m.chukin@mail.ru; pavel_poletskov@mail.ru; olganikitenko@list.ru; nabat4ikov@mail.ru***

AUTHOR'S INFO

**M. V. Chukin**, Dr. Eng., Prof., Head of the Chair of Material Processing Technology, First Vice-Rector on Science and Innovations,
**P. P. Poletskov**, Dr. Eng., Prof., the Chair of Material Processing Technology,
**O. A. Nikitenko**, Cand. Eng., Research Assistant,
**D. G. Nabatchikov**, Cand. Eng., Research Assistant

*Key words:*

low-carbon pipe steel, rolled heavy planes, microalloying, controlled rolling, accelerated cooling, microstructure, cold resistance, acicular ferrite, quasi-polygonal ferrite, bainite-martensite areas

ABSTRACT

The results of investigations of microstructure and properties of heavy plates made of cold-resistant pipe steel are presented. The series of laboratorial melting experiments on low-carbon steel has been undertaken in the conditions of "Termodeform-MGTU" researching complex with variation of content of alloying elements. Physical simulation of controlled rolling with accelerated cooling according to different procedures has been conducted during this work.

Mechanical testing of rolled products have been conducted according to the standard techniques with determination of strength and cold resistance (amount of tough component in the fracture of samples for drop weight tear testing (DWTT) at the temperature –55 °C.

The complex of metallograhic investigations has been undertaken in the Centre of collective use (TsKP) of the Scientific and research institute of Nanosteels at Nosov Magnitogorsk State Technical University. Rolled microstructure has been investigated by the methods of optical and scanning electron microscopy. It was revealed that forming of the mixture containing acicular ferrite, polygonal (polyhedral) ferrite and quasi-polygonal ferrite is the optimal way with relation to providing of high cold resistance (> 90 % of tough component in the fracture of samples for DWTT). It was noted that presence in microstructure of essential part of bainite with different morphology forming along the boundaries of large austenite grains leads to deterioration of the properties of rolled material during drop weight tear testing.

## Introduction

Rise of the strength parameters, impact toughness and cold resistance with simultaneous improvement of material weldability is considered as the development direction for low-carbon microalloyed pipe steels. At the same time demand for rolled products with increased thickness increases; it is required for manufacture of tubes with high operating pressure and for welded construction [1]. Reliability of such constructions is based, according to the up-to-date vision, on braking of extended tough destruction. To provide it, essential rise of impact toughness of the main metal used in constructions (especially at the temperatures of the upper threshold of cold brittleness) is required [2]. Resistance of large-diameter tubes to extended brittle destructions is guaranteed in the case of getting the positive results during drop weight tear testing (DWTT) with providing the part of tough component in the fracture of samples not less than 85%.

Combination of strength and tough-ductile properties, required for rolled heavy plates, can be achieved in the modern metallurgical practice by forming ferrite-bainite microstructure of rolled material with fine grain size (matrix element) and increased density of dislocations [1]. This ferrite-bainite microstructure of steel can be obtained on the condition of providing of austenite overcooling: introduction of the elements rising austenite stability (Mo, Cr, Ni, Cu) in steel and usage of accelerated cooling of rolled products [3, 4].

The technology of controlled rolling with consequent accelerated cooling includes subsequent refining of steel microstructural elements at the all process stages [5]. Intensive refining of austenite grain with its multiple complete recrystallization occurs during the roughing stage of controlled rolling. As soon as possibilities of grain refining during recrystallization are restricted, creation of increased density of defects of crystalline construction (being the place of origination of α-phase during transformation) is provided by substantial summarized deformation (usually not less than 70%) of non-recrystallized austenite during the finishing stage of rolling [4, 6]. Accelerated cooling provides essential overcooling of austenite, what results in $\gamma\rightarrow\alpha$ transformation and rise of speed of origination of new grains and, consequently, increase of grain size is restricted. Forming of dispersed products via the shift transformation mechanism instead of products of pearlite transformation occurs during phase transformation of overcooled austenite [1].

To provide forming the fine-grained ferrite-bainite microstructure, low-alloyed pipe steel should have low carbon content (0,04–0,07%), increased manganese content (≤1,95%), additives of the elements rising austenite stability (Mo, Cr, Ni, Cu) and complex microalloying by carbonitride-forming elements Nb+Ti (+V)) ≤ 0,15% [7, 8].

Lowered carbon content provides niobium transition in solid solution during slab heating before rolling (it is re-

* The work has been conducted with participation of S. V. Denisov, P. A. Stekanov, E. V. Braichev, N. V. Koptseva, Yu. Yu. Efimova, M. S. Gushchina, A. S. Ishimov, M. S. Zherebtsov, D. M. Chukin.

Table 1. **Chemical composition of the examined steel**

| No. of sample | Mass part,% not more or in the range | | | | | | | | Other elements | $C_{eq}$ | $P_{cm}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | C | Si | Mn | P | S | Cr | Ni | V | | | |
| 1 | 0.05–0.07 | 0.30 | 1.95 | 0.007 | 0.004 | 0.30 | 1.00 | 0.048 | Cu, Mo, Nb, Ti | 0.55 | 0.23 |
| 2 | | | | | | 0.30 | 1.00 | 0.048 | | 0.56 | 0.23 |
| 3 | | | | | | 0.05 | 1.00 | 0.054 | | 0.52 | 0.22 |
| 4 | | | | | | 0.30 | 1.00 | 0.049 | | 0.55 | 0.22 |
| 5 | | | | | | 0.05 | 1.00 | 0.051 | | 0.52 | 0.21 |
| 6 | | | | | | 0.05 | 1.00 | 0.050 | | 0.48 | 0.20 |
| 7 | | | | | | 0.30 | 1.00 | 0.051 | | 0.54 | 0.22 |
| 8 | | | | | | 0.05 | 1.00 | 0.042 | | 0.50 | 0.21 |
| 9 | | | | | | 0.05 | 1.00 | 0.043 | | 0.51 | 0.22 |
| 10 | | | | | | 0.05 | 0.20 | 0.043 | | 0.44 | 0.19 |
| 11 | | | | | | 0.05 | 1.00 | 0.001 | | 0.49 | 0.20 |
| 12 | | | | | | 0.05 | 1.00 | 0.003 | | 0.49 | 0.20 |

Remark. Calculation of carbon equivalent $C_{eq}$ и $P_{cm}$ is conducted via the formulas:

$$C_{eq} = C + \frac{\mathrm{Mn}}{6} + \frac{\mathrm{Cr+Mo+V}}{5} + \frac{\mathrm{Cu+Ni}}{15}; \quad P_{\mathrm{cm}} = C + \frac{\mathrm{Mn+Cr+Cu}}{20} + \frac{\mathrm{Si}}{30} + \frac{\mathrm{Ni}}{60} + \frac{\mathrm{Mo}}{15} + \frac{\mathrm{V}}{10} + 5\,\mathrm{B}$$

Table 2. **Procedures of thermomechanical treatment**

| No. of sample | Heating temperature, °C | Thickness of rolled semi-product, mm | Interphase cooling | Initial temperature of the 2nd phase, °C | Finishing temperature of accelerated cooling, °C |
|---|---|---|---|---|---|
| 1 | 1200 | 165 | Air cooling | 890 | 520–560 |
| 2 | 1120 | | Air cooling | 930 | |
| 3 | 1120 | | Air cooling | 930 | |
| 4 | 1200 | | Water cooling | 860 | |
| 5 | 1200 | | Water cooling | 860 | |
| 6 | 1200 | | Water cooling | 860 | |
| 7 | 1200 | | Water cooling | 860 | |
| 8 | 1200 | | Air cooling | 860 | |
| 9 | 1200 | | Water cooling | 930 | |
| 10 | 1200 | | Air cooling | 860 | |
| 11 | 1200 | | Air cooling | 860 | |
| 12 | 1200 | | Air cooling | 860 | |

quired to expand the area where austenite recrystallization does not occur and, respectively, to work out steel structure) and facilitates improvement of steel impact toughness and weldability [1]. Part of niobium that did not extract during rolling in the austenite area leaves in solid solution and rises austenite stability in $\gamma \rightarrow \alpha$ transformation; it also promotes forming of ferrite-bainite microstructure [3, 6]. Lowered carbon content and high steel purity for harmful impurities ($S \leq 0{,}002\%$, $P \leq 0{,}015\%$) provide good cold resistance and weldability of metal.

The technology of controlled rolling with accelerated cooling for manufacture of cold-resistant rolled products with large thickness (41 mm and more) has its features. The main of them are technological problems connected with deformation of metal with essential thickness using high partial reduction as well as temperature gradient during accelerated cooling with consequent redistribution of these temperatures across sections of the plates [1]. Austenite decomposition in the modern low-alloyed steels can occur during phase transformation with forming of wide spectrum of products of intermediate (bainite) transformation with non-polyhedral morphology [9–12]. Violations of the production technology lead usually to forming of non-optimal microstructure of rolled products, what finalizes first of all in deterioration during drop weight tear testing of metal cold resistance as the most structurally sensitive steel property [13–16].

The innovative technology for manufacture of ultra cold-resistant nanostructured rolled plates is developing in the framework of realization of the complex project aimed on creation of the high-tech production facilities. Heavy plates manufactured by this technology will be used to provide import substitution of materials, including cryogenic materials used in the conditions of extra low critical temperatures, increased corrosion activity and also in Arctic regions. The above-mentioned complex project is realizing be the initiative of Magnitogorsk Iron and Steel Works with participation of Nosov Magnitogorsk State Technical University and "Termodeform-MGTU" researching complex.

The aim of this work is investigation of the relationship between microstructure and DWTT parameter for rolled heavy planes made of cold-resistant steel for manufacture of large diameter electric-welded and longitudinal-welded tubes.

## Material and technique of investigation

The ingots of low-carbon alloyed steel with consequent varying of chromium, nickel and vanadium content have been melt in "Termodeform-MGTU" researching complex (**tab. 1**).

Reduction of ingots has been conducted in accordance with the procedures of controlled rolling, using the hydraulic press. The ingots with size 300×100×60 mm have been preliminary heated up to the temperature 1120–1200 °C (**tab. 2**). Afterwards the ingots have been subjected to upsetting down to 165 mm, and the further interphase cooling has been realized via two technologies: in air or using simulating plant for controlled water cooling in combination with press (**fig. 1**). Then the ingots have been reduced to the size 41×100×400 mm. The total amount of passes is 9–11, with the degree of partial deformations from 5 to 15%. The obtained rolled semiproducts have been subjected to accelerated cooling from 760–800 °C to 520–560 °C with different cooling rates in the range 4–15 m/s.

**Fig. 1. Technological process of thermomechanical treatment**

Table 3. **Mechanical properties of the examined steel**

| No. of sample | Yield strength $\sigma_т$, N/mm² | Relative elongation, $A_{50}$,% | Tensile strength $\sigma_в$, N/mm² | Part of tough component in fracture of DWTT samples at testing temperature – 55 °C,% |
|---|---|---|---|---|
| 1 | 590 | 35 | 655 | 0 |
| 2 | 580 | 40 | 680 | 0 |
| 3 | 580 | 45 | 670 | 0 |
| 4 | 525 | 55 | 720 | 90 |
| 5 | 570 | 50 | 670 | 50 |
| 6 | 520 | 55 | 670 | 95 |
| 7 | 530 | 50 | 690 | 85 |
| 8 | 540 | 58 | 670 | 90 |
| 9 | 565 | 45 | 660 | 35 |
| 10 | 510 | 63 | 590 | 100 |
| 11 | 515 | 57 | 640 | 95 |
| 12 | 590 | 50 | 700 | 0 |

Extension testings have been implemented on longitudinal samples at room temperature according to ASTM A370. Testings for impact bending (DWTT test) has been conducted at the vertical impact tension machine DWT 40-5 at the temperature –55 °C according to API RP 5L3 (**tab. 3**). The results of fractures of DWTT samples are presented on the **fig. 2**.

The complex of metallographic investigations has been executed in the Centre of collective use (TsKP) of the Scientific and research institute of Nanosteels at Nosov Magnitogorsk State Technical University. The polished sections have been prepared from samples according to the standard technique for microanalysis. This preparation has used pressing of samples in resin "Transoptic" at the automatic press Simplimet 1000 of Buechler company.

Surface of polished section has been conducted to pickling in 4% solution of nitric acid in ethyl alcohol via dipping of polished surface in the bath with reagent.

To reveal qualitative and quantitative parameters of forming structure, the optical microscope Meiji Techno with usage of computer analysis system Thixomet PRO for images has been applied. The most typical pictures of microstructure for 1/4 of rolled plate thickness with DWTT parameters 0%, 35–50% и 95–100% are presented on the **fig. 3**.

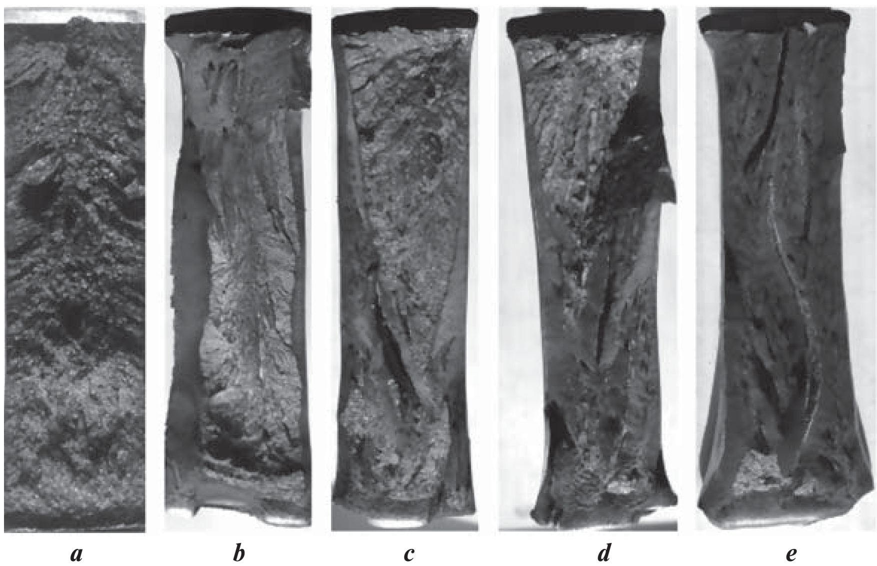

**Fig. 2. The most typical fractures of DWTT samples:**
*a* — 0%, *b* — 35%, *c* — 50%, *d* — 85%, *e* — 100%

Microstructure has been examined using scanning electron microscopy JSM 6490 LV in secondary electrons and with magnification more than 1000 times (**fig. 4**). Microhardness has been determined on hardness meter Buchler Mikromet via impression of a diamond pyramid with angle among the opposite planes 136° and in correspondence with GOST 9450-60 for load 1 kg and loading duration 10 s.

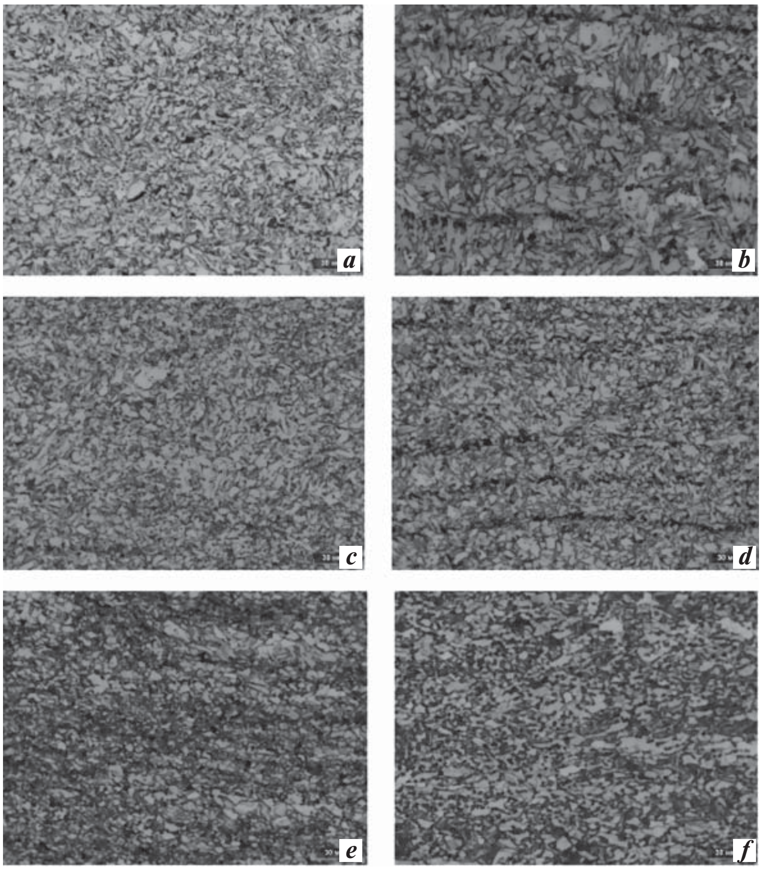


**Fig. 3. Optic microstructure of steel, ×500:**
*a, b* — 0% DDTW; *c* — 35%; *d* — 50%; *e* — 85%; *f* — 100%

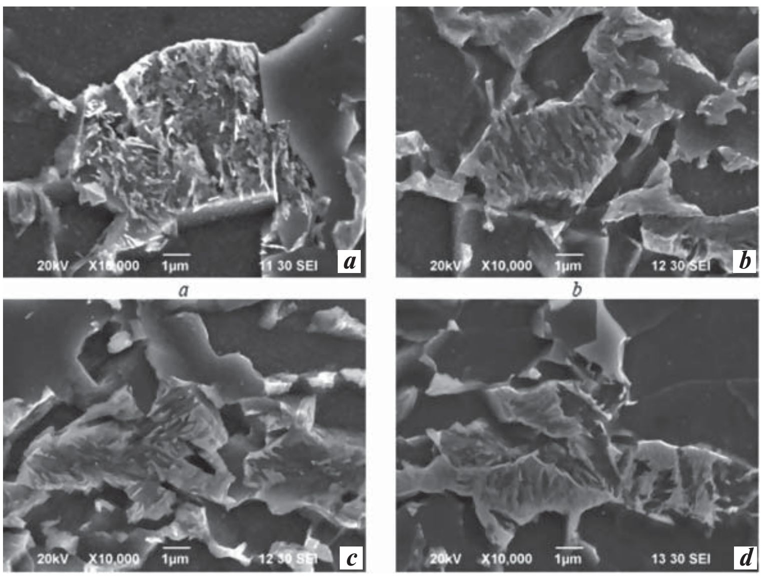


**Fig. 4. Microstructure of rolled plates (scanning electron microscope), bainite-martensite areas (BMA):**
*a, b* — sample № 2; *c, d* — sample № 3

**Results of investigation and their discussion**

Quantitative analysis of microstructure has been conducted in order to determine relationships between structural components and cold resistance of rolled plates (**table 3**), and then the obtained results have been compared with the part of tough component in fracture of DWTT samples.

Microstructure of the samples No. 1, 2, 3, 12 (0% DWTT) is presented by 100% bainite. In the central part and on the surface we observe acicular ferrite with the islands of bainite-martensite component along grain boundaries; they have been revealed using scanning electron microscopy. The grain form is non-equiaxial and angular.

Microstructure of the samples No. 1, 2, 3, 12 (85–100% DWTT) is presented by the mixture of acicular ferrite (AF), granular ferrite (GF), polygonal (polyhedral) ferrite (PF) and quasi-polygonal ferrite (QPF). The carbon-containing phase is presented by degenerating pearlite (DP) consisting of dispersed mixture of ferrite, low-temperature pearlite and upper bainite (UB). Dispersed islands from DP+UB mixture are located on the boundaries of grains QPF and blocks GF and AF. The grain form is irregular (crooked) with slightly expressed sub-grain structure and increased density of dislocations.

Microstructure of the samples No. 5 and 9 (35–50% DWTT) is presented by the mixture of acicular ferrite (AF) up to ~50% and quasi-polygonal ferrite (QPF) with relatively small number of the islands of carbon-containing phase along the grain boundaries. The grain form is non-equiaxial and angular.

Thereby combination of high strength and cold resistance for rolled heavy plates with large thickness can be provided via forming of dispersed steel microstructure consisting of acicular and quasi-polygonal ferrite. Presence of substantial part of bainite with different morphology in microstructure leads to deterioration of the properties of rolled product during drop weight tear testing.

**Conclusion**

Analysis of obtained results of metallographic investigations of low-carbon microalloyed steel after thermomechanical treatment testifies about the conclusion that high operating properties of pipe metal are provided by forming of dispersed steel microstructure consisting of acicular and quasi-polygonal ferrite. Presence of substantial part of bainite with different morphology in microstructure leads to deterioration of cold resistance and, as a result, to decrease of the part of tough component in fracture of DWTT samples.

Table 3. **Quantitative parameters of microstructure of the examined steel**

| No. of sample | Average diameter ($d_{av}$) of ferrite grains, μm | | Average length ($l_{av}$) of ferrite (bainite) aciculas, μm | | Volumetric part ($V$) of acicular ferrite (bainite) / (BMA),% | |
|---|---|---|---|---|---|---|
| | 1/4 h* | surface | 1/4 h* | surface | 1/4 h* | surface |
| 1 | 5.43 | 2.82 | 15.3 | 7.52 | 65.37 / 8.63 | 65.1 / 12.2 |
| 2 | 4.44 | 3.89 | 11.3 | 9.3 | 58.3 / 10.9 | 80.9 / 10.1 |
| 3 | 5.03 | 6.66 | 3.72 | 7.35 | 79.5 / 6.5 | 92 / 0 |
| 4 | 8.29 | 2.83 | 18.6 | 5.52 | 50.7 / 12.5 | 53 / 7 |
| 5 | 4.42 | – | 7.69 | (12.1) | 76.43 / 8.8 | 93.58 / 6.42 |
| 6 | 4.14 | 2.38 | – | 8.22 | - / 8.56 | 8.6 / 7 |
| 7 | 3.42 | 3.26 | 7.29 | 10.2 | 11.5 / 7.78 | 98 / 2 |
| 8 | 4.23 | 3.41 | 6.08 | 9.54 | 4 / 5.61 | 100 / 0 |
| 9 | 4.67 | 3.53 | 8.87 | 9.57 | 10 / 2 | 100 / 0 |
| 10 | 6.43 | 5.85 | (7.24) | (6.46) | (20) / 0 | (25) / 0 |
| 11 | 4.22 | 3.03 | 8.32 | 6.18 | 84.5 / 4.5 | 8 / 4 |
| 12 | 3.78 | 3.56 | 8.23 | 9.18 | 89 / 10 | 95.5 / 4.5 |

* h — thickness of rolled product

***The study was financially supported by Ministry of Education and Science of the Russian Federation within the scope of accomplishment of multiple-purpose project on creation of modern high-tech production with the participation of higher education institution (Contract 03.G25.31.0235).***

## REFERENCES

1. Nastich S. Yu., Morozov Yu. D., Matrosov M. Yu., Denisov S. V., Galkin V. V., Stekanov P. A. Implementation of production facilities for rolled heavy plates made of low-alloyed steels with increased strength and cold-resistance pdrdmeters at 5000 rolling mill of Magnitogorsk Iron and Steel Works. *Metallurg*. 2011. No. 11. pp. 57–64.
2. Kantor M. M., Bozhenov V. A. Material science provision of durability of major pipelines. *Stal*. 2010. No. 4. pp. 80–84.
3. Heisterkamp F., Hulka K., Matrosov M. Yu. et al. Niobium-containing low-alloyed steels. Moscow : Intermet Engineering, 1999. 94 p.
4. Matrosov M. Yu., Efron L. I., Ilyinsky V. I. et al. Usage of accelerated coolingfor rising of mechanical and technological properties of rolled heavy plates for manufacture of large diameter gas pipeline tubes. *Metallurg*. 2005. No. 6. pp. 49–54.
5. Salganik V. M., Poletskov P. P., Berezhnaya G. A., Gushchina M. S., Alekseev D. Yu. Investigation of the effect of deformation degree on austenite grain size in low-alloyed steel. *Proizvodstvo prokata*. 2016. No. 3. pp. 3–8.
6. DeArdo A. J. Modern Thermomechanical Processing of Microalloyed Steel: A Physical Metallurgy Perspective: *Microalloying'95 Conf. Proc. (Pittsburgh, PA, June 11–14, 1995.)*. The Iron and Steel Society. pp. 15–33.
7. Chukin M. V., Poletskov P. P., Denisov S. V., Gushchina M. S., Berezhnaya G. A., Nikitenko O. A., Ishimov A. S. Influence of chromium on structure, phase transformations and properties of high-strength steels. *Proizvodstvo prokata*. 2016. No. 8. pp. 14–18.
8. Chukin M. V., Poletskov P. P., Nabatchikov D. G., Gushchina M. S., Berezhnaya G. A. Influence of alloying elements on steel properties for different cooling rates. *Estestvennye i tekhnicheskie nauki*. 2016. No. 8. pp. 62–65.
9. Krauss G., Thompson S. W. Ferritic Microstructures in Continuous Cooled Low- and Ultralowcarbon Steels. *ISIJ Int*. 1995. Vol. 35. No 8. pp. 937–945.
10. Smirnov M. A., Pyshmintsev I. Yu., Boryakova A. N. On the problem of classification of microstructure of low-carbon pipe steels. *Metallurg*. 2010. No. 7. pp. 45–51.
11. Matrosov M. Yu., Efron L. I., Kichkina A. A., Lyasotsky I. V. Study of microstructure of pipe steel microalloyed by niobium after different procedures of controlled rolling with accelerated coolong. *Metallovedenie i termicheskaya obrabotka metallov*. 2008. No. 3 (633). pp. 44–49.
12. Li Jia-D., Jiang Z.-H., Li S.-P. et al. Continuous Cooling Transformation Behaviors and Mechanical Properties of High Strength Microalloyed Pipeline Steel. *Proc. HSLP 2010 Int. Seminar on Application of High Strength Line Pipe 2010 (June 28–29, 2010. Xi'an, China)*. pp. 126–133.
13. Nastich S. Yu. Influence of morphology of the bainite component of microstructure of low-alloyed steel X70 on cold-resistance of rolled plates with large thickness. *Metallurg*. 2012. No. 3. pp. 62–69.
14. Kim Y. M., Kim S. K., Lim Y. J. et al. Influence of Microstructure on Yield/Tensile Strength Ratio and Low-Temperature Toughness of Pipeline Steels. *ISIJ Int*. 2002. Vol. 42. No. 12. pp. 1571–1577.
15. Morozov Yu. D., Goli-Oglu E. A., Nastich S. Yu., Knyshev A. V. Modern approaches to production of low-carbon microalloyed pipe steel at Steckel mills. Chernye metally. 2012. No. 9. pp. 8–14.
16. Kazakov A. A., Kazakova E. I., Kiselev D. V., Motovilina G. D. Development of evaluation methods for microstructural heterogeneity of pipe steels. Chernye metally. 2009. No. 12. pp. 12–15.