

Features of forming the structural and phase state of low-alloy steel for flexible tubing pipes depending on thermokinetic transformation conditions

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Demand in Russia for flexible tubing pipes (FTP) increased during last decades by 80 %, and import substitution of foreign technologies in FTP production became a priority task after introduction of sanctions. Coiled rolled sheet of high-strength low-alloy steel with increased resistance to atmospheric corrosion is used for FTP manufacture. The operating features in the North regions determine necessity of using for FTP the materials meeting the requirement of ST80 strength grade according to the standard API Specification 5ST: tensile strength ≥ 610 MPa, relative elongation ≥ 20 %, Rockwell hardness ≤ 22 HRC and increased cold resistance down to -60 °C. The paper describes the research of the forming features of structural and phase state of low-alloy steel for FTP with ST80 strength grade, having the following original chemical composition, % (mass.), not exceeding: 0.16C; 0.50Si; 1.00Mn; 0.70Cr; 0.80(Ni+Cu+Mo); 0.050(Nb+V+Ti), depending on thermokinetic transformation conditions. Structural and thermokinetic diagrams were built for the new steel with original chemical composition. To provide the required complex of properties on the base of the selected thermokinetic transformation conditions, the parameters of controlled rolling with accelerated post-deformation cooling were determined. Based on the results of investigations, it was established that achievement of the highest level of viscous-plastic properties ($KCV^{-60} = 122$ J/cm²) at the preset strength level of the steel with ST80 strength grade ($\sigma_u \geq 610$ MPa, HRC ≤ 22) is provided in steel owing to forming of dispersed ferrite-bainite structure. This process occurs during realization of controlled phase transitions: the temperature of the beginning and the end of accelerated cooling 830 °C and 600 °C respectively.

Key words: flexible tubing pipes, low-alloy steel, ST80 strength grade, thermokinetic diagram, controlled rolling with accelerated cooling, ferrite-bainite structure, hardness.

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Introduction

Demand in Russia for flexible tubing pipes (FTP) increased during last decades by 80 %, what is caused by putting into operation and mastering of the new deposits and drilling technologies in the oil and gas industry. USA and China are the global leaders in FTP production. All FTP requirements of oil and gas companies were satisfied only via import, and at present time, after introduction of sanctions, it increases the risks for companies and makes import substitution of foreign technologies the priority task [1, 2]. It is known that coiled rolled sheet of high-strength low-alloy steel of the type 4 (according to the international standard ASTM A606) is used. This standard presents the requirements of the material with increased resistance to atmospheric corrosion in its chemical composition and mechanical properties [3–7].

Additionally, the problem of developing the deposits located at high latitudes with complicated mining and geological structure, i.e. in the hardly accessible Far North and Arctic regions, deserves special attention [8]. The operating features in the north regions stipulate necessity of using materials for FTP, which at the same time meet the requirements of the international standard API Specification 5ST «Specification for Coiled Tubing» [9]. The steels

for FTP with ST80 strength grade are considered as such materials. They are characterized by the following properties: tensile strength ≥ 610 MPa, yield strength within the range 550–620 MPa, relative elongation ≥ 20 %, Rockwell hardness HRC ≤ 22 ; they also have increased cold resistance at the temperature down to -60 °C. Achieving of such parameters will provide possibility if using metal products in the Arctic climatic conditions [1, 5].

Microstructure is the most important factor having influence on the properties of coiled rolled products; its forming is stipulated by steel chemical composition (1) and by thermal and deformation processing (2) [10–12]. Thermomechanical processing, in particular controlled rolling [13, 14] is widely used at present time for effective grain refinement and providing a high level of steel mechanical properties, i.e. viscous and plastic parameters. Controlled rolling includes billet heating, preliminary deformation at the temperature above the austenite recrystallization temperature and final deformation in the area without austenite recrystallization (or in the intermediate $\gamma+\alpha$ area) and cooling, what allows obtaining austenite structure with high density of structural defects in the end of rolling [13].

It should be noted that the most homogenous and dispersed are structure components, and the closer they are to

the state of thermodynamic equilibrium, the most resistant is steel to the effects of aggressive media [15–19]. The structures of ferrite-pearlite type, which are forming in low-alloy steels during cooling at low speeds, may have pearlite stripes as nodes of cracks forming and propagation. Analysis of cracked areas displays that cracks are forming either in pearlite itself, or just close to a separation boundary between ferrite and pearlite [20, 21]. Ferrite-bainite structure is obtained during accelerated cooling in the process of thermomechanical processing; it is characterized by rather smaller banding and size of ferrite matrix elements, what has favourable effect on the properties of hot-rolled coiled products [22].

Revealing the features of forming the structural and phase state of low-alloy steel for FTP, depending on thermokinetic transformation conditions for selection of the best parameters of accelerated cooling in the process of thermomechanical treatment, is the aim of this research.

Material and methods of the research

The research was carried out with laboratorial samples of the steel with ST80 strength grade having the following original chemical composition, % (mass.), not exceeding: 0.16C; 0.50Si; 1.00Mn; 0.70Cr; 0.80(Ni+Cu+Mo); 0.050(Nb+V+Ti). Melting, continuous casting and thermomechanical processing of this steel were conducted using the equipment of scientific and production complex “Termodeform-MGTU” of Nosov Magnitogorsk State Technical University.

Dilatometric testing of steel samples were carried out using the Pocket Jaw module of the complex Gleeble 3500. Heating of the samples with diameter 6–10 mm up to austenite state was implemented in vacuum up to the temperature 930 °C with rate 3.3 °C/s via direct current passage. Consequent cooling was conducted at the rates within the range from 1 to 100 °C/s.

Metallographic researches were carried out in the Centre of common use at “Nanosteels” Scientific and research institute of Nosov Magnitogorsk State Technical University. Polished sections for microanalysis were fabricated according to the standard technique in the Buehler sample preparation line.

A light microscope Zeiss Axio Observer, using the software Thixomet PRO for processing of metallographic images as well as via scanning electron microscope (SEM) JSM 6490 LV were used for microstructure analysis. Quantitative analysis was carried on light and electron microscope microstructure images using the software complex Thixomet PRO.

The complex of mechanical tests was carried out according to the special techniques. Testing for hardness of the samples was conducted according to the GOST 2999-75 via indenting of a diamond pyramid with the angle 136° between opposite planes, with load 9.807 N (1 kgf) and loading duration 10 s.

Results and discussion

Critical points in the examined steel, which were determined via bends on dilatometric curves, were the following: $A_{c1} \approx 688$ °C, $A_{c3} \approx 807$ °C. Analysis of these curves displayed that ferrite extraction from austenite as well as pearlite, bainite and martensite transformations are observed during continuous controlled cooling (Table 1).

The results of optical microscopy are presented in the Fig. 1. Metallographic analysis displayed that hypoeutectic ferrite is observed in microstructure of the examined steel for all cooling rates; its amount decreases from 80 % to 15 % with increase of cooling rate from 1 to 100 °C/s. It should be noted that starting from the rate 15 °C/s, the part of ferrite increases slightly.

For more detailed identification of structural components and revealing features of their morphology, the SEM investigation was carried out (Fig. 2).

Electron and microscope analysis displayed that the sample cooled with the rate 1 °C/s, includes pearlite with fine-dispersed structure in addition to ferrite (Fig. 2a); hardness value is equal to 165 HV.

When the cooling rate is 5 °C/s, the structure includes, in addition to ferrite and small amount of pearlite, also granulated bainite (appr. 33 %) containing of α -phase with martensite-austenite sections inside it (Fig. 2b). Presence of bainite component in microstructure leads to hardness increase to 198 HV.

Table 1. Temperatures of phase transformations for the examined steel during continuous cooling

| Cooling rate, °C/s | Temperatures of phase transformations, °C | | | | | | |
|-----------------------|---|-------------------------|--------|------------------------|--------|---------------------------|--------|
| | Ferrite extraction | Pearlite transformation | | Bainite transformation | | Martensite transformation | |
| | Start | Start | Finish | Start | Finish | Start | Finish |
| 1 | 767 | 618 | 570 | – | – | – | – |
| 5 | 758 | 590 | 572 | 562 | 503 | – | – |
| 10 | 744 | – | – | 569 | 495 | – | – |
| 15 | 741 | – | – | 568 | 478 | – | – |
| 20 | 736 | – | – | 568 | 464 | – | – |
| 30 | 732 | – | – | 552 | 430 | 430 | 330 |
| 40 | 726 | – | – | 543 | 435 | 435 | 337 |
| 60 | 713 | – | – | 510 | 434 | 434 | 330 |
| 80 | 705 | – | – | 480 | 435 | 435 | 330 |
| 100 | 693 | – | – | 468 | 439 | 439 | 311 |

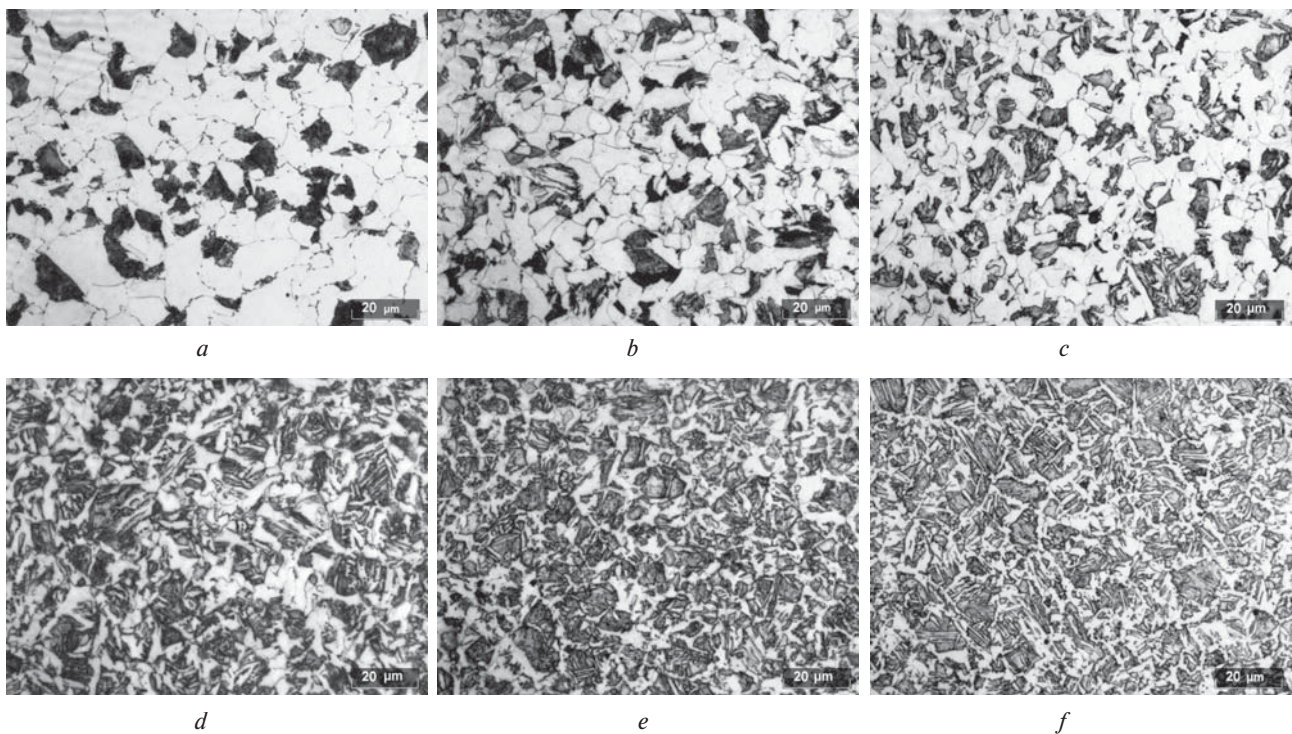


Fig. 1. Steel microstructure after cooling with the rates, °C/s: 1 (*a*), 5 (*b*), 10 (*c*), 15 (*d*), 30 (*e*) and 100 (*f*), $\times 1000$

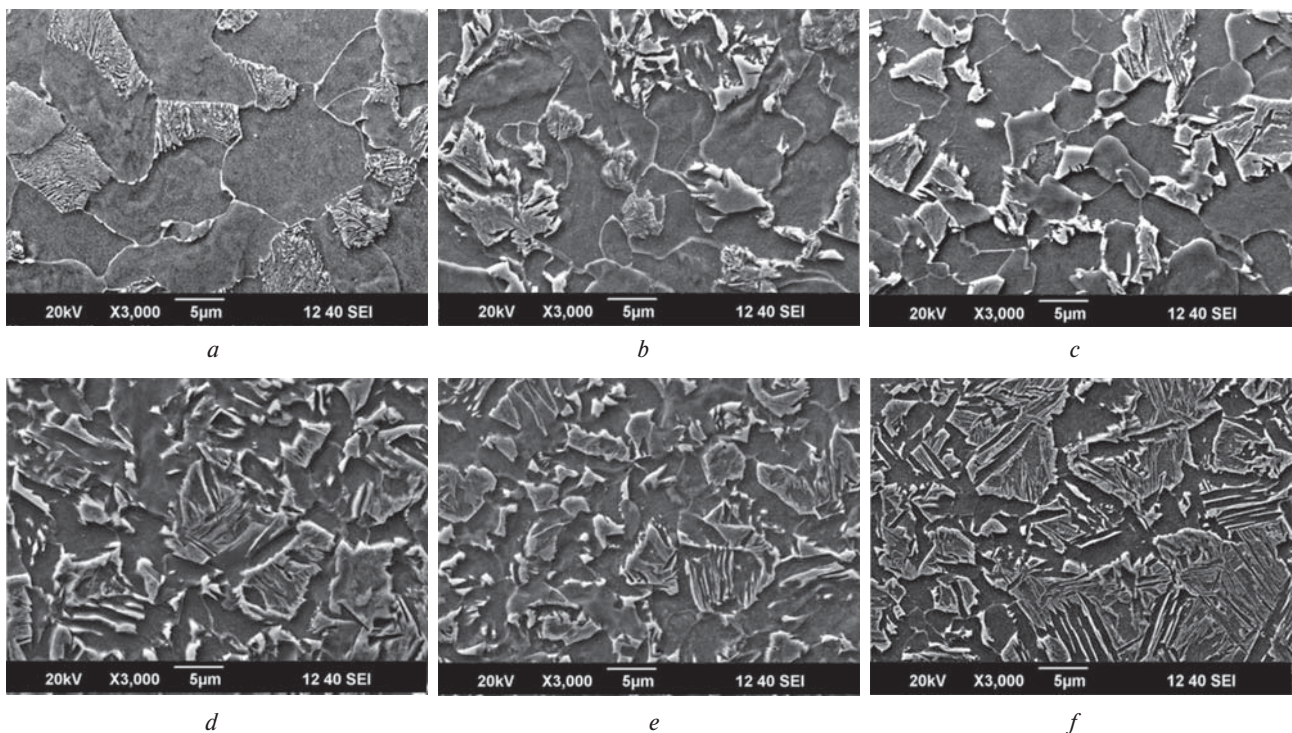


Fig. 2. Features of building of steel structural components after cooling with the rates, °C/s: 1 (*a*), 5 (*b*), 10 (*c*), 15 (*d*), 30 (*e*) and 100 (*f*)

When the cooling rate is 10 °C/s, pearlite is not revealed in microstructure; it testifies on suppression of diffusion pearlite transformation (Fig. 2c). Bainite and quasi-polygonal ferrite, which is located mainly as a net along the boundaries of bainite areas, are identified in microstructure; it is observed at the cooling rates 15 and 20 °C/s (Fig. 2c and 2d). The relative part of bainite increases in this case up to 80 %, what leads to hardness rise to 237 HV.

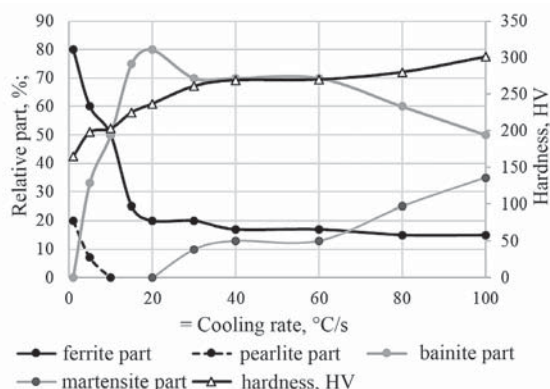
Starting from the cooling rate is 30 °C/s, martensite appears in microstructure (Fig. 2e). When the cooling rate rises up to 100 °C/s, the amount of ferrite and bainite reduces to 15 % and 50 % respectively, while the amount of martensite increases to 35 % (Fig. 2f), what is accompanied by hardness rise to 301 HV.

The results of quantitative analysis of microstructure and the hardness values after cooling with various rates are presented in the **Table 2**.

Table 2. Quantitative parameters of microstructure and the hardness values of the examined steel

| Cooling rate, °C/s | Relative volumetric part of structural components, % | | | | Hardness, HV1 |
|--------------------|--|----------|---------|------------|---------------|
| | Ferrite | Pearlite | Bainite | Martensite | |
| 1 | 80 | 20 | 0 | 0 | 165 |
| 5 | 60 | 7 | 33 | 0 | 198 |
| 10 | 50 | 0 | 50 | 0 | 203 |
| 15 | 25 | 0 | 75 | 0 | 225 |
| 20 | 20 | 0 | 80 | 0 | 237 |
| 30 | 20 | 0 | 70 | 10 | 261 |
| 40 | 17 | 0 | 70 | 13 | 269 |
| 60 | 17 | 0 | 70 | 13 | 270 |
| 80 | 15 | 0 | 60 | 25 | 280 |
| 100 | 15 | 0 | 50 | 35 | 301 |

Interpretation of the complex dilatometric and metallographic analyses as well as hardness measurements allowed to build the structural diagram reflecting relationship between volumetric part of structural components and cooling rate (Fig. 3). Thermokinetic diagram (TKD) of decomposition of overcooled austenite (Fig. 4) for the steel having the following original chemical composition, % (mass.), not exceeding: 0.16C; 0.50Si; 1.00Mn; 0.70Cr; 0.80(Ni+Cu+Mo); 0.050(Nb+V+Ti) was built as well.

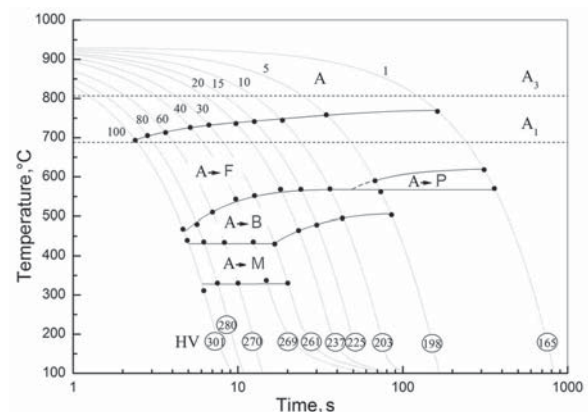
**Fig. 3. Relationship between volumetric part of structural components and hardness of the examined steel from the cooling rate**

The experience of FTP production shows that high-strength steel for FTP manufacture is characterized by low relationship between yield strength and tensile strength during extension and should have multi-phase microstructure containing mainly of bainite and quasi-polygonal ferrite [6, 22–24]. According to the thermokinetic diagram of the examined steel, such structure is forming within the cooling rate range 10–30 °C/s. Additionally, the hardness value should not exceed 22 HRC / 250 HV, according to the requirements for FTP steels of ST80 strength grade [1, 5, 25]. This hardness should be provided at the cooling rates not exceeding 20–25 °C/s, in correspondence with the thermokinetic diagram (see Fig. 4).

Based on the obtained data, different procedures of controlled rolling were proposed and realized using the equipment of scientific and production complex “Termodeform-MGTU”, with heating up to 1200 °C in order to obtain homogenous austenite and with varying the temperatures of

the beginning and finishing of accelerated cooling as well as the cooling rate. As a result, the following rolling procedures were tested:

- finishing of deformation and starting of accelerated cooling in the single-phase austenite γ -area ($A_{r3} + (10–20)^\circ\text{C}$);
- finishing of deformation and starting of accelerated cooling in the single-phase austenite $\gamma + \alpha$ -area ($A_{r3} + (30–50)^\circ\text{C}$);
- providing of accelerated cooling rate for obtaining ferrite-bainite structure not exceeding $\sim 25^\circ\text{C/s}$;

**Fig. 4. Thermokinetic diagram of transformation of overcooled austenite, % (mass.), not exceeding: 0.16C; 0.50Si; 1.00Mn; 0.70Cr; 0.80(Ni+Cu+Mo); 0.050(Nb+V+Ti): A = austenite; F = ferrite; P = pearlite; B = bainite; M = martensite**

- selection of the temperature of finishing of accelerated cooling based on the condition of obtaining the required complex of properties.

Based on the results of mechanical testing of laboratory samples of the steel having the following original chemical composition, % (mass.), not exceeding: 0.16C; 0.50Si; 1.00Mn; 0.70Cr; 0.80(Ni+Cu+Mo); 0.050(Nb+V+Ti), it was established that achieving of the highest level of viscous and plastic properties ($KCV_{-60} = 122 \text{ J/cm}^2$) at the preset strength level of the steel with ST80 strength grade ($\sigma_u = 583 \text{ MPa}$, $\sigma_{0.2} = 723 \text{ MPa}$, HRB 95 (HRC < 20)) is provided after controlled rolling with accelerated post-deformation cooling. This procedure is conducted according to the following procedure: temperature of the beginning of accelerated cooling 830°C and the temperature of its finishing 600°C . Metallographic examinations displayed that the

required dispersed ferrite-bainite structure was obtained in steel (Fig. 5). High dispersity of structural components after such treatment is caused by austenite grain refining during roughing rolling and obtaining of austenite with high density of defects during finishing rolling (within the temperature range below recrystallization temperature, but above the temperature of the beginning of $\gamma + \alpha$ -transformation).

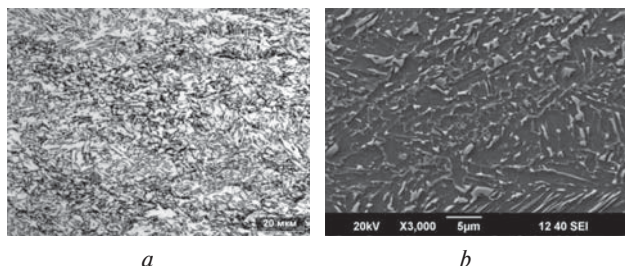


Fig. 5. Steel microstructure after controlled rolling with accelerated cooling: *a* — light microscopy, $\times 1000$; *b* — scanning electron microscopy, $\times 3000$

Conclusion

Regularities of the phase and structural transformations during continuous cooling of the high-strength steel for FTP with ST80 strength grade, having the following original chemical composition, % (mass.), not exceeding: 0.16C; 0.50Si; 1.00Mn; 0.70Cr; 0.80(Ni+Cu+Mo); 0.050(Nb+V+Ti), were established. Based on the obtained data for the steel with original chemical composition, the thermokinetic diagram of overcooled austenite decomposition is built.

Based on the determined thermokinetic transformation conditions, the parameters of controlled rolling with accelerated cooling were suggested. Observation of these parameters provides achieving the properties corresponding to the steels for FTP with ST80 strength grade, due to forming of dispersed ferrite-bainite structure during implementation of controlled phase transitions.

It was revealed that achieving of the highest level of viscous and plastic properties ($KCV^{-60} = 122 \text{ J/cm}^2$) at the pre-set strength level of the steel with ST80 strength grade ($\sigma_u \geq 610 \text{ MPa}$, $HRC \leq 22$) is provided in the examined steel after controlled rolling with accelerated post-deformation cooling at the following parameters: temperature of the beginning of accelerated cooling 830°C and the temperature of its finishing 600°C .

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