Metallurgist, Vol. 57, Nos. 1–2, May, 2013 (Russian Original Nos. 1–2, Jan.–Feb., 2013)

IMPROVEMENT OF CHEMICAL COMPOSITION AND PRODUCTION REGIMES FOR MANUFACTURE OF K65–K70 (X80–X90) STRIP BASED ON SIMULATION

A. A. Kruglova, V. V. Orlov,
O. V. Sych, and E. I. Khlusova

UDC 621.77:669.14.018.29:621.643

The effect of deformation temperature scheme in the finish rolling stage and cooling regime on structural component morphology and set of strength properties for pipe steel is demonstrated. Improved chemical composition and production regimes are established on the basis of quantitative correlations for structure and properties of pipe steel using a Gleeble simulation system. Results are presented for manufacture of industrial test batches of rolled sheet of strength classes K65–K70 (X80–X90).
Keywords: *simulation, Gleeble 3800 system, thermomechanical treatment, alloy composition, finish rolling stage, accelerated cooling end temperature, cooling rate, bainite, ferrite, substructure, strength, brittle failure resistance.*

In 2007–2010 as applied to Severstal Cherepovets Metallurgical Combine (CherMK) equipment manufacturing technology was developed for rolled sheet for pipes of strength classes K65 (X80) – K80 (X100) based on manganese-nickel composition alloy steel [1]. The results obtained made it possible to provide manufacture of domestic strip and the for the first time to provide full-scale import substitution of high technology product. In 2009, a set of equipment of the 5000 mill was started up at Magnitogorsk Metallurgical Combine (MMK). This is new generation equipment with a high degree of automation, requiring fundamentally new scientific approaches and study methods in developing production schemes for manufacturing rolled sheet.

The principles of structure formation under industrial conditions should take account of the technical characteristics of equipment used (rolling mill power, position of supplementary equipment, type of cooling system, possibility of computer management of process parameters). Here limitations for rolling power parameters of contemporary 5000 mills govern requirements for the production process:

1) with a high torsional moment in rolls (rolling moment) combined with a low rolling force, development of the structure should be provided predominantly in the roughing stage;

2) with high force and limited rolling momen,t development of a metal structure is accomplished more rapidly in the finishing stage.

The aim of the work is to choose chemical composition, rolling temperature and deformation parameters, and cooling conditions, making it possible to obtain a unique combination of required pipe steel in relation to production potential of the MMK equipment.

However, industrial experiments have become more and more expensive. The Gleeble 3800 system makes it possible to simulate thermomechanical treatment (TMT) for steel. Variation of key TMT parameters, i.e., frequency of reduction in finishing and roughing stages, degree and temperature of deformation in each pass, deformation rate, cooling temperature and rate, makes it possible to improve the chemical composition and thermodynamic rolling and cooling regimes for low-alloy

Prometey Central Research Institute of Structural Materials (TsNII KM Prometey), St. Petersburg, Russia; e-mail: victorm@crism.ru.
Translated from Metallurg, No. 2, pp. 50–58, February, 2013. Original article submitted October 15, 2012.

TABLE 1. Test Steel with C_e = 0.44 Alloy Composition

Steel alloying version	Chemical composition, wt.%						
	C	Mn	Ni	Cr	Cu	Mo	Microalloying
Composition No. 1	0.06	1.70	Up to 0.20	Up to 0.20	+	+	Nb + V + Ti
Composition No. 2			Up to 0.40	–	+	+	Nb + Ti

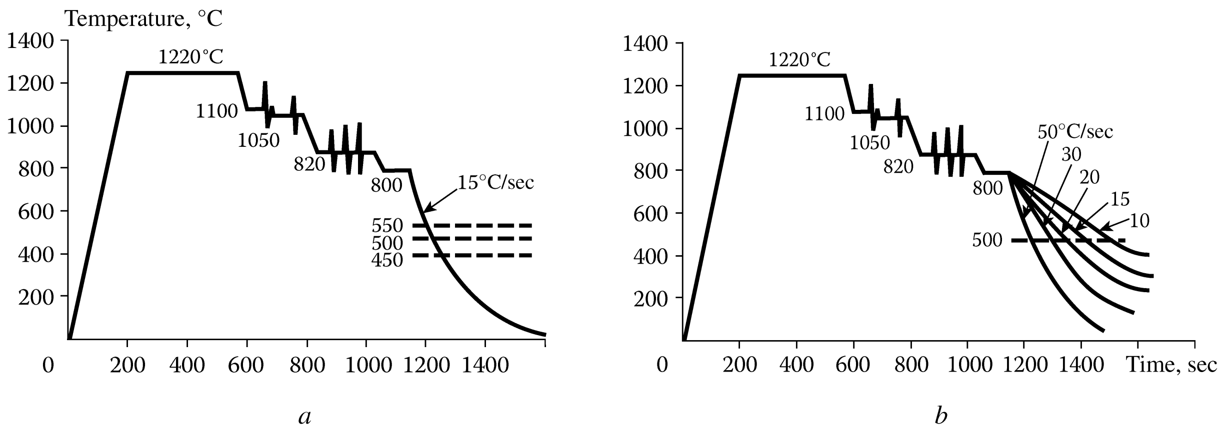

Fig. 1. TMT scheme for determining the optimum cooling regime in a Gleeble system by variation: *a*) accelerated cooling end temperature; *b*) cooling rate.

low-carbon steels of strength classes K65–K70 (X80–X90), providing a reduction in cost, an increase in productivity, and manufacturing economic efficiency. In view of this, in order to achieve the required mechanical properties and finely dispersed structure, choice of chemical composition and thermodynamic parameters for the finish rolling stage and accelerated cooling (AC) for high-strength pipe steel have been accomplished on the basis of simulation modelling.

The material selected for the study was manganese pipe steel of two basic alloy systems with different chromium, nickel, and vanadium content (Table 1). The carbon equivalent was calculated using the equation $C_e = C + Mn/6 + (Cr + Mo + V)/5 + (Ni + Cu)/15$.

Simulation of TMT in a Gleeble 3800 system was carried out on cylindrical specimens 10 mm in diameter and 15 mm high with deformation by compression. In order to determine strength properties tests were performed in compression for cylindrical specimens with a size of 6 × 10 mm cut from deformed billets. The yield and ultimate strengths were established by calculation, proceeding from the similarity of true deformation curves with uniaxial compression and tension.

In order to select the optimum TMT regimes, cooling and the finish rolling stage scheme were varied (Figs. 1 and 2). The temperature for the end of AC was varied within the limits 450–550°C (Fig. 1*a*); the cooling rate was 10–50°C/sec (Fig. 1*b*).

In order to determine the optimum, three deformation regimes were tested in the finishing stage: with a steady temperature of 820°C (No. 1), from decreasing 840→800°C (No. 2), and increasing 800→840°C (No. 3) temperature curves (see Fig. 2).

On the basis of studies by means of the Gleeble 3800 system, industrial testing was carried out for manufacturing technology of rolled sheet strength classes K65 (X80) and K70 (X90) by the recommended regimes.

Evaluation of mechanical properties was carried out in accordance with specifications of the standard ISO 3183–2007. Metal brittle failure resistance was determined from results of drop weight tear testing of full-scale samples with a test temperature of –20°C with determination of the amount of ductile component is a specimens fracture for DWTT according to API 5L3 (*B*(DWTT), %).

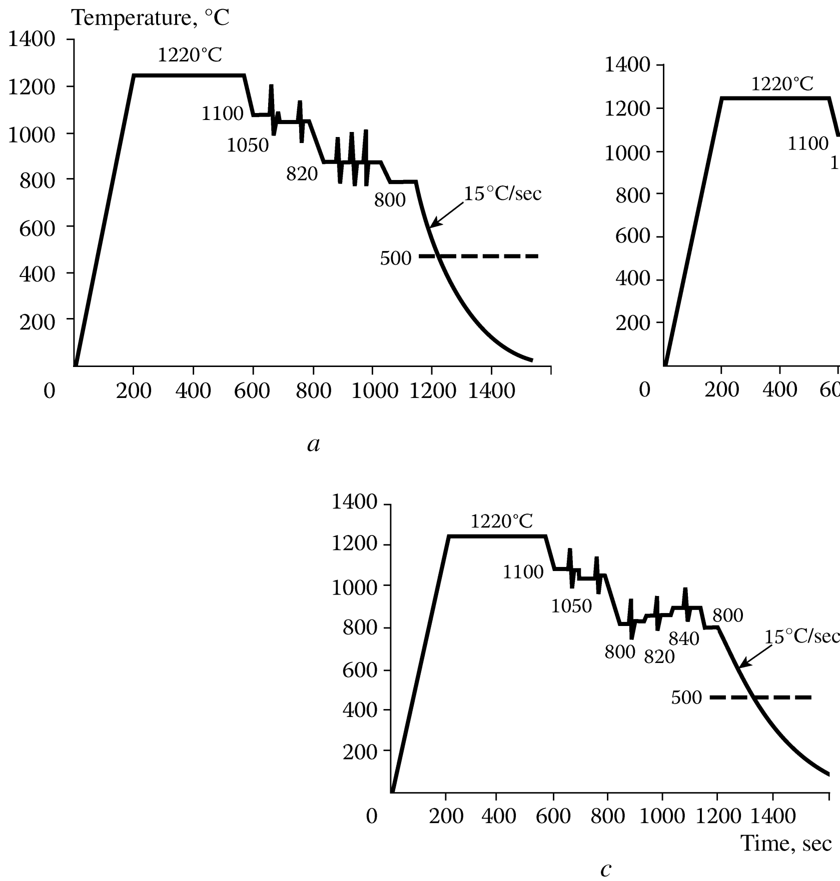

Fig. 2. TMT scheme for determining the finish rolling stage optimum regime in a Gleeble 3800 system: *a*) deformation by regime No. 1; *b*) deformation regime No. 2; *c*) deformation by regime No. 3.

Microstructures were studied by optical metallography in microsections after etching in 3% alcoholic HNO_3 solution according to GOST 5640. A quantitative estimate of the content of ferrite, granular and lath bainite after different TMT regimes was determined by means of a Thixomet image analyzer and a Quanta 3D FEG scanning electron microscope by automation electron back-scattered diffraction (EBSD) analysis, and in fact analysis of interphase misorientations.

Simulation modelling in a Gleeble 3800 system. Formation of structure with a different ration and dispersion of structural components (polygonal ferrite and bainite of granular and lath morphology) with variation of temperature for the end of cooling, cooling rate, and deformation scheme without changing alloy composition, makes it possible to measure the set of strength and ductility properties satisfying the steel classes from X70 (K60) to X90 (K70).

Studies made it possible to establish that:

for composition No. 1, σ_u = 640–800 MPa, σ_y = 460–650 MPa;
for composition No. 2, σ_u = 680–800 MPa, σ_y = 500–650 MPa.

As is well known, formation within a structure of excess ferrite leads to a reduction in steel strength with an increase in toughness and ductility, predominance of a lath type structure, i.e., to an increase in strength properties with a simultaneous reduction in ductility and brittle failure resistance in finished rolled sheet [2–5]. Therefore, the optimum combination of mechanical properties is achieved with formation in rolled sheet of steel strength class: K60 (X70), a ferrite-bainite structure with proportion of free ferrite up to 50%; for K65 (X80), a ferrite-bainite structure with bainitic granular morphology and proportion of ferrite up to 25–30%; for K70 (X90), predominantly bainitic structure of lath and granular morphology.

TABLE 2. Effect of Cooling End Temperature on Strength Properties (σ_y MPa/σ_u MPa) for Pipe Steel

Steel alloy version	End of accelerated coolingtemperature, °C		
	450	500	550
Composition No. 1	543/755	505/682	462/648
Composition No. 2	625/773	570/710	530/685

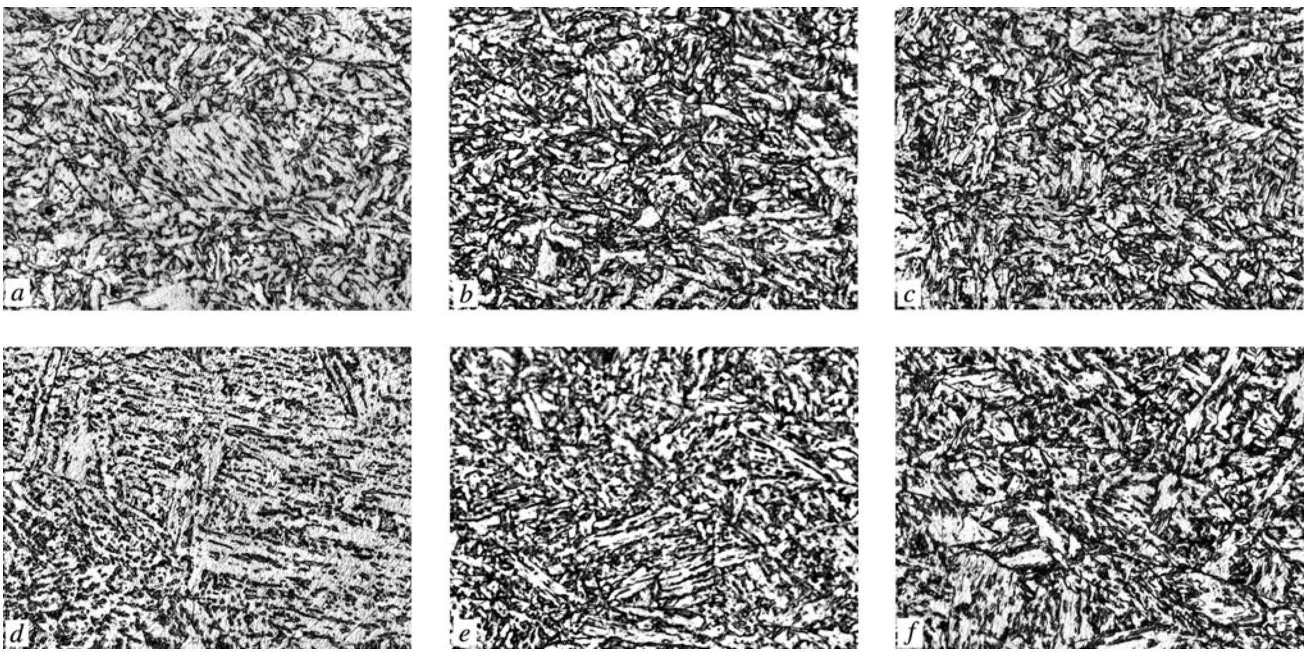

Fig. 3. Effect of cooling rate on the type of structure for pipe steel of compositions No. 1 (*a–c*) and No. 2 (*d–f*) with end of cooling temperature: *a*, *d*) 450°C; *b*, *e*) 500°C; *c*, *f*) 550°C; ×500.

Choice of temperature for the end of accelerated cooling. Results of determining yield and ultimate strengths for specimens after simulating TMT with cooling to temperatures of 450, 500, and 550°C at a rate of 15°C/sec (see Fig. 1*a*) are presented in Table 2.

An increase in temperature for the end of cooling leads to an increase in the amount of free ferrite and granular bainite (Fig. 3). *After cooling to 550°C* in steels of both compositions there is formation of a ferrite-bainite structure. Formation of granular morphology bainite and free ferrite in an amount of 30–40% in steel of composition No. 1 (see Fig. 3*c*) promotes a reduction in σ_y value to 460–470 MPa and σ_u to 640–650 MPa. In steel of composition No. 2 after cooling to 550°C a mixed structure forms with bainite of granular and lath morphology and proportion of free ferrite of the order of 20% (see Fig. 3*f*), which makes it impossible to obtain a value of σ_y higher than 520–530 MPa and σ_u above 680–690 MPa.

A reduction for the end of cooling to 450°C promotes formation in steel composition No. 1 of an inhomogeneous ferrite-bainite structure with coarse areas of bainite of lath morphology (see Fig. 3*a*), and σ_y = 540–550 MPa and σ_u = = 750–760 MPa, in steel of composition No. 2 there is a bainite structure of lath morphology (see Fig. 3*d*) which promotes an increase in σ_y to 625 MPa and σ_u = 770–780 MPa.

The optimum structure in test steels without excess free ferrite, reducing strength, lath bainite, reducing viscoplastic properties, is formed after cooling to 500°C (see Fig. 3*b*, *e*), and for compositions Nos. 1 and 2 σ_y = 505 and 570 MPa, σ_u = 680 and 710 MPa, respectively.

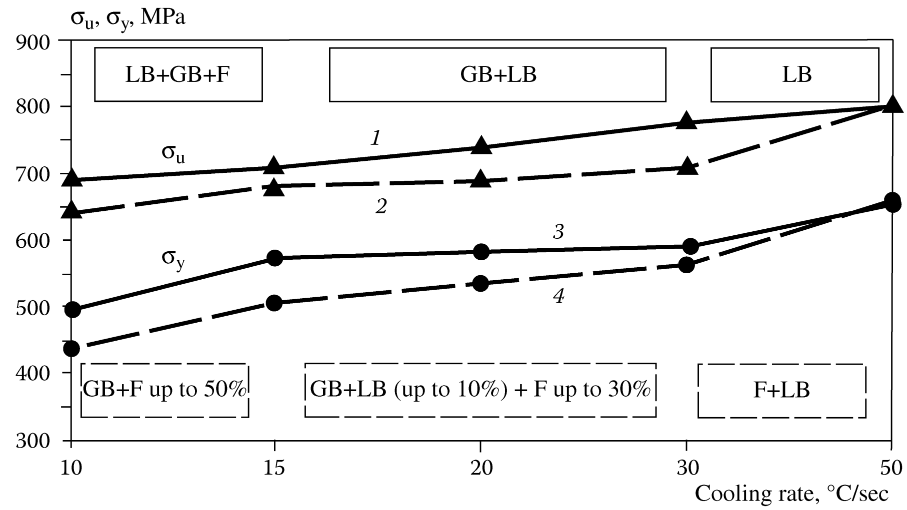

Fig. 4. Dependence of strength properties and structure type (GB is granular bainite, LB is lath bainite, F is ferrite) on cooling rate: *1*, *3*) composition No. 2; *2*, *4*) composition No. 1.

Choice of cooling rate. The effect of cooling rate to 500°C after TMT on the structure and strength properties of steel compositions Nos. 1 and 2 (Fig. 4) was studied.

Over the whole test range of cooling rates (10–50°C/sec) in steel of composition No. 1 there is formation of a ferrite-bainite structure (Fig. 5*a*, *b*). With an increase in cooling rate there is a change in bainite morphology from granular to lath, and amount of ferrite. With cooling rates of not more than 15°C/sec there is formation of bainite of granular morphology with a proportion of ferrite of the order of 40–50%, σ_y = 445–500 MPa, σ_u = 650–660 MPa. An increase in cooling rate to 15–30°C/sec promotes an increase in strength properties (σ_y up to 520–570 MPa, σ_u up to 660–700 MPa) as a result of a reduction in the amount of free ferrite to 30% and formation alongside bainite of granular morphology (60%) of some amount of lath bainite (up to 10%). With cooling rates above 30°C/sec in steel of composition No. 1 transformation commences with precipitation of free ferrite at grain boundaries of former austenite grains, within whose limits there is formation of areas of lath bainite. Here σ_u = 750–800 MPa. σ_y = 600–650 MPa.

In steel of composition No. 2, with higher nickel content, over the whole test range of cooling rates there is formation of a predominantly bainitic structure, which is caused by the effect of nickel on reduction in phase transformation temperature. With cooling rates less than 15°C/sec in steel of composition No. 2 there is formation of granular bainite structure and ferrite in an amount up to 15–20% with σ_u = 690–700 MPa and σ_y = 495–550 MPa. Formation of bainite of granular and lath morphology with a cooling rate of 15–30°C/sec promotes an increase in strength properties: σ_u = 720–770 MPa, σ_y = 570–590 MPa. Cooling at a rate above 30°C/sec leads to formation in steel of composition No. 2 of lath bainite with σ_u = 780–800 MPa, σ_y = 625–650 MPa.

A study of the structure with the use of EBSD analysis, in particular, for estimating the relative extent of boundaries with different misorientation angles, confirmed the different ratio of structural components in relation to cooling rate (see Fig. 5). Generally for steel with a ferrite-bainite structure there is typically presence of two ranges of misorientation angles between grain boundaries: 5–15° and 50–60°. In the first of them there are so-called low-angle boundaries ($\Theta < 15°$), the main part of which is connected with the bainite component, and in the second, there are large-angle boundaries ($\Theta \geq 15°$), connected with the ferrite component of the structure. With cooling rates not more than 15°C/sec for steels of both compositions there is typically presence of a considerable number of large-angle boundaries with misorientation angles up to 60° (extent of the order of 65–70% of the overall extent of all boundaries), which points to predominant formation of ferrite and granular bainite. The proportion and extent of low-angle boundaries is not more than 20–25% (see Fig. 5*a*, *d*). With cooling rates of 15–30°C/sec, formation of predominantly bainitic structure with development of a sub-structure is confirmed by preferential formation of

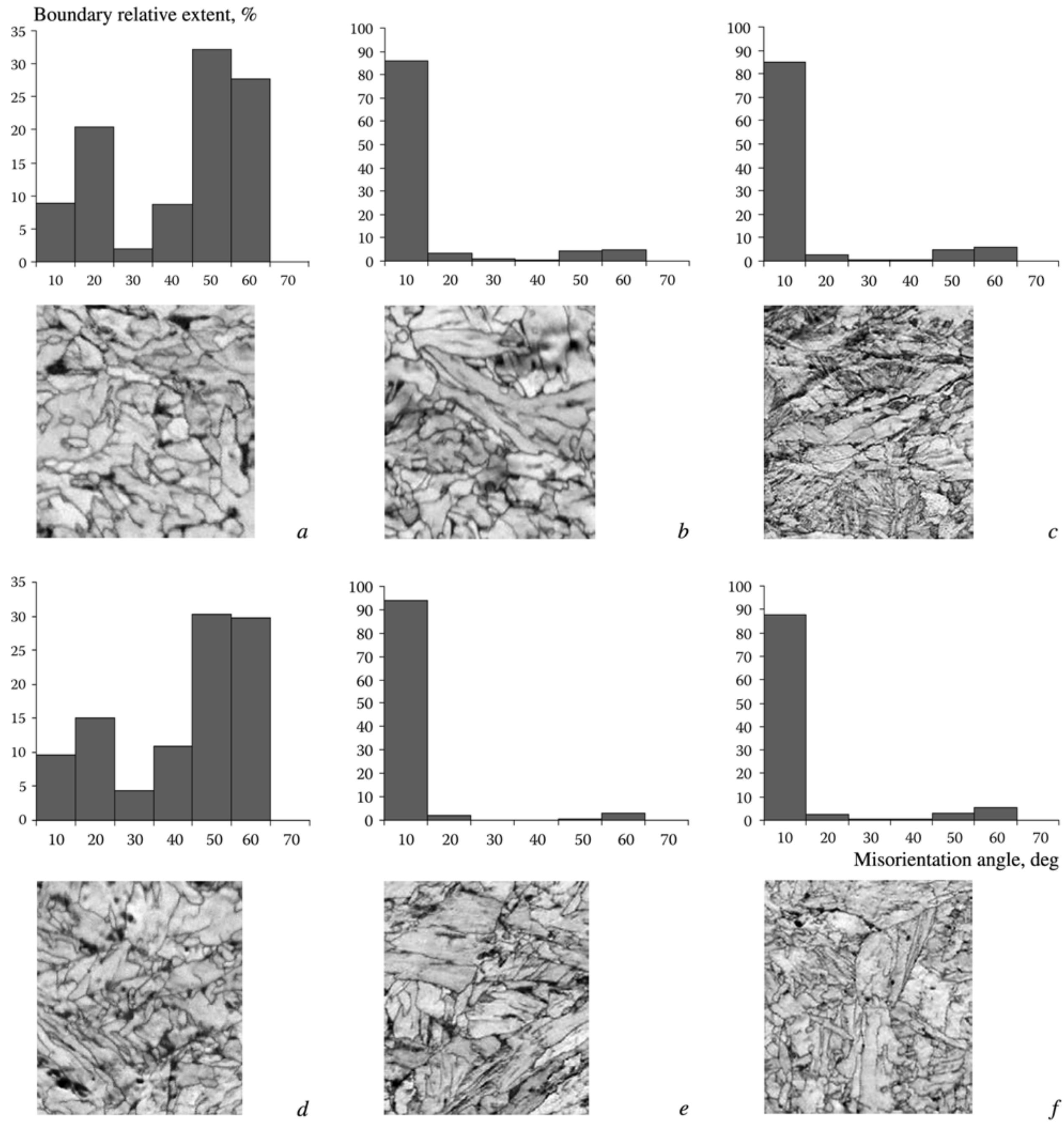

Fig. 5. Effect of cooling rate on the morphology and fineness of structural components for pipe steel of compositions No. 1(*a*–*c*) and No. 2 (*d*–*f*) in relation to cooling rate: *a*, *d*) 10°C/sec; *b*, *e*) 20°C/sec; *c*, *f*) 50°C/sec.

low-angle boundaries with their relative extent: of the order of 85% for composition No. 1 (see Fig. 5*b*), and 95% for composition No. 2 (see Fig. 5*e*). Formation of a bainitic structure of predominantly lath morphology after cooling at a rate of 50°C/sec is confirmed by an increase in the amount of low-angle boundaries (see Fig. 5*c*, *f*).

Thus, on the basis of the structure–ultimate strength correlation the optimum range of 15–30°C/sec was selected for cooling rates of pipe rolled sheet with which there is formation:

TABLE 3. Effect of Finish Rolling Stage Temperature on Strength Properties (σ_y MPa/σ_u MPa) for Pipe Steel

Steel alloying version	Deformation in finish rolling stage		
	Regime 1	Regime 2	Regime 3
Composition No. 1	505/682	518/697	525/712
Composition No. 2	570/710	595/748	635/795

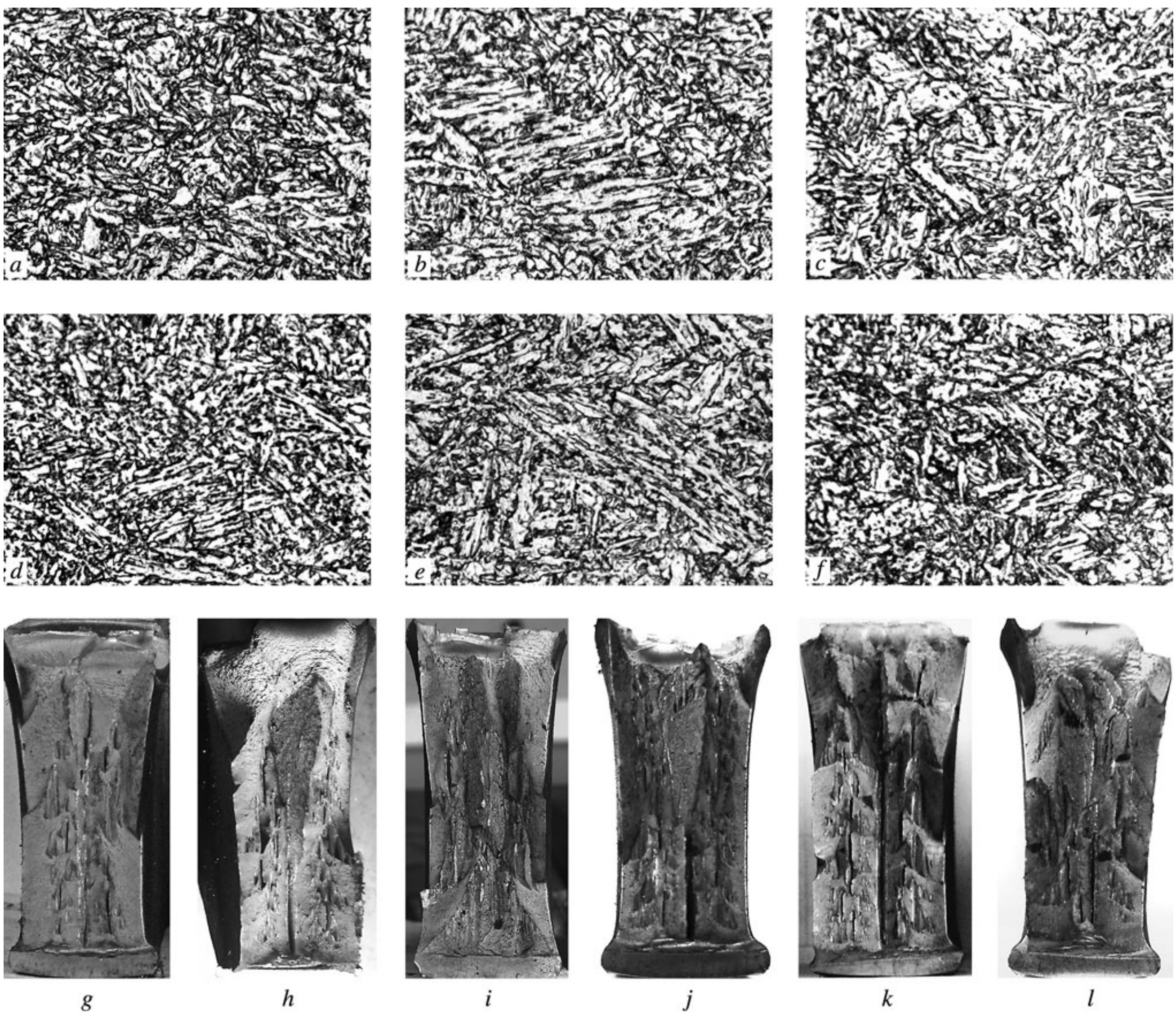

Fig. 6. Effect of the finish rolling stage temperature regime on the structure, ×500, (*a–f*) and the form of sample fracture after DWTT (*g–l*) for pipe steel of compositions No. 1 (*a–c*, *g–i*) and No. 2 (*d–f*, *j–l*): *a*, *d*, *g*, *j*) deformation by regime 1; *b*, *e*, *h*, *k*) deformation by regime 2; *c*, *f*, *i*, *l*) deformation by regime 3.

in steel of composition No. 1 of a bainite-ferrite structure with bainite predominantly of granular morphology. Lath bainite in an amount up to 10% and proportion of free ferrite up to 30% with the level σ_u = 680–700 MPa, satisfying specifications for this property for rolled sheet of strength class K65 (X80), i.e., not less than 650 MPa;

in steel No. 2 there is predominantly a bainitic structure of granular and lath morphology with a higher σ_u = 720–780 MPa compared with composition No. 1 satisfying the specification of this property for steel of strength class K70 (X90), i.e., nor less than 695 MPa.

Choice of finish rolling stage temperature regime. The main tasks of two-stage TMT for rolled sheet of high-strength pipe steels are:

1) preparation of a finer austenite grain size with multistage reduction not less than 14% per pass in the roughing rolling stage;

2) formation of an austenite structure with high dislocation density and developed sub-structure in the finish rolling stage, inherited by the transformed structure.

In view of the equipment features of the MMK 5000 mill, development of an austenite structure is accomplished predominantly in the finish rolling stage as a result of significant plastic deformation at a high rate.

Previous studies, including those the Prometei Research Institute established [6] that performance of the finish stage at below 1000°C (in the region of retarded austenite recrystallization) leads to formation after cooling of an inhomogeneous structure, characterized by presence of coarse areas of lath bainite and ferrite along boundaries of former austenite grains, and this has a negative effect on the content of fibrous component in fractures of large samples with drop weight tear testing. The optimum temperature range for performing the roughing rolling stage is 1100–1050°C.

The finish rolling regime should provide formation of the maximum possible amount of centers for grain generation in order to form a fine structure with accelerated cooling [7]. The possibility of varying deformation rate up to 5 m/sec makes it possible to control the structure and properties due to realizing different thermal deformation schemes for the finish rolling stage, both isothermal, and increasing (with heating of rolled product with high degrees of reduction and deformation rates) or a decreasing curve in a narrow temperature range.

Results of determining σ_y and σ_u of specimens after TMT simulation by different schemes for the finish rolling stage after cooling to 500°C with a rate of 15°C/sec are presented in Table 3.

With a change in temperature scheme for the finish rolling stage (regimes 1→2→3) there is an increase in steel strength properties as a result of increasing the amount of lath bainite (regime 3) or austenite work hardening (regime 2).

In *steel of composition No. 1* with variation of the temperature scheme for finish rolling strength properties change little (by 20–30 MPa) in spite of the fact that after treatment by regimes 2 and 3 there is formation predominantly of lath bainite (Fig. 6*b*, *c*), and after treatment by regime 1 it is granular (see Fig. 6*a*). This may be explained by the high degree of dispersion of ferrite-bainite structure, formed after TMT with deformation in the finish stage at a constant temperature of 820°C, providing *B*(DWTT) = 95% (Fig. 6*g*). Deformation by regime 2 promotes formation alongside granular bainite of coarse areas of lath bainite (see Fig. 6*b*), reducing *B*(DWTT) to 85% (Fig. 6*h*). After deformation by regime 3 there is formation of a ferrite-bainite structure, having high fineness for bainite of granular and lath morphology with presence of individual coarse ferrite grains of irregular shape (see Fig. 6*c*), which also has an unfavorable effect on brittle failure resistance (*B*(DWTT) = 85%) (Fig. 6*i*).

In *steel of composition No. 2* after deformation by regimes 1 and 2 there is formation of a finely dispersed predominantly bainitic structure of granular and lath morphology (Fig. 6*d*, *e*). An increase in σ_y by 25 MPA and σ_u by 38 MPa after deformation in the finish stage by a decreasing temperature curve 840 → 800°C is caused by an increase in the degree of austenite work hardening, inherited by bainitic ferrite (Fig. 6*f*). After deformation by regime 2 *B*(DWTT) = 95% (Fig. 6*k*). After deformation by regime 3 there is formation of lath bainite with a high degree of extension of structural components along the deformation direction, providing an increase in σ_y to 635 MPa, σ_u to 795 MPa with a simultaneous reduction in brittle failure resistance (*B*(DWTT) = 80%) (Fig. 6*l*).

Thus, on the basis "structure – ultimate strength, proportion of ductile component" correlation a temperature regime was selected for deformation in the finish rolling stage for pipe steel:

for composition No. 1, deformation with a constant temperature of 820°C provides σ_u = 680–690 MPa and *B*(DWTT) = 95%, satisfying the specifications for rolled sheet of strength class K65 (X80);

for composition No. 2, deformation by a decreasing temperature curve 840 → 800°C provides σ_u = 740–750 MPa, *B*(DWTT) = 95%, satisfying specifications for rolled sheet of strength class K70 (X90).

TABLE 4. Rolled Sheet Mechanical Properties of Steel Strength Classes K65–K70 (X80–X90)

Alloy version / strength class	In transverse direction, min–max/average					
	σ_y, MPa	σ_u, MPa	δ_5, %	σ_y/σ_u	KCV^{-40}, J/cm^2	B(DWTT), %
Composition No. 1 / K65 (X80)	575–650/610	665–730/695	20–24.5/21	0.84–0.92/0.88	270–450/370	95–100/97.5
Composition No. 2 / K70 (X90)	630–680/655	710–760/735	19–22.5/20	0.86–0.92/0.89	270–420/335	90–97.5/92

Development of recommendations and approval of industrial production of rolled sheet of strength classes K65–K70 (X80–X90). Thus, analysis of features established by means of simulation modelling in a Gleeble 3800 system for correlation of structure and properties and existing testing of rolled sheet of strength classes K65–K70 (X80–X90) as applied to CherMK equipment made it possible to improve the chemical composition and temperature-deformation parameters of TMT for the automated MMK 5000 mill:

1) for production of rolled sheet of strength class K65 (X80), Mn–Ni–Cr–Mo–V–Nb–Ti-steel (composition 1) is recommended, providing high ductility and brittle failure resistance with required strength level;

2) for production of rolled sheet of strength class K70 (X90), Mn–Ni–Cu–Nb–Ti-steel (composition 2) is recommended with a reduction to 0.44% for steel of this strength class of the carbon equivalent, providing high strength and required of ductility, and brittle failure resistance.

The improved production regimes have been proven in manufacture of rolled sheet of strength classes K65–K70 (X80–X90) at the MMK 5000 mill, whose level of mechanical properties is presented in Table 4.

Conclusions

1. By means of contemporary methods for simulation modelling and study of the structure the effect has been established for temperature schemes in the finish rolling stage and accelerated cooling regimes (temperature and rate) on the type, fineness, and morphology of structural components and set of mechanical properties for pipe steel.

2. It has been shown that in order to obtain a set of properties corresponding to strength class K65 (X80) it is desirable to use steel Mn–Ni–Cr–Mo–V–Nb–Ti alloy composition providing an increase in critical points for Ar_3 and Ar_1 transformations. This makes it possible, combined with an isothermal deformation scheme in the finish rolling stage, as a result of a high plastic deformation rate with reductions not less than 12% per pass and cooling at a rate of the order of 15–20°C/sec to the temperature of the middle region of the bainitic range, to form in steel predominantly granular bainite and quasipolygonal ferrite (in an amount up to 30%) in order to increase the stability of viscoplastic properties and brittle failure resistance.

3. In order to obtain a set of properties for pipe steel corresponding to strength class K70 (X90), it is desirable to use steel of Mn–Ni–Cu–Nb–Ti alloy composition with a higher nickel content, providing a reduction in phase transformation temperatures. This makes it possible combined with deformation in the finish rolling stage according to a decreasing curve in quite a narrow temperature range with reduction not less than 12% per pass, promoting austenite work hardening, and cooling at a rate of 20–30°C/sec to the temperature of the middle of the bainitic range, to form predominantly bainite of granular and lath morphology with high dislocation density and developed sub-structure, inherited by bainitic ferrite with $\gamma \to \alpha$-transformation.

4. Realization of the principles established for structure formation and properties made it possible to improve the chemical composition and production parameters for manufacturing rolled sheet of strength classes K65–K70 (X80–X90) for pipes of main pipelines with the aim of increasing stability of operating characteristics and reducing pipe steel cost.

REFERENCES

1. E. Kh. Shakhpazov, Yu. D. Morozov, S. Yu. Nastich, et al., "High-strength steels for main pipelines," *Vopr. Material.*, No. 3, 127–138 (2009).

2. Yu. D. Morozov, M. Yu. Matrosov, S. Yu. Nastich, and A. B. Arabei, “High-strength pipe steel of a new generation with a ferrite-bainite structure,” *Metallurg*, No. 8, 39–42 (2008).
3. M. Yu. Matrosov, L. I. Efron, V. I. Il’inskii, et al., “Use of accelerated cooling for improving mechanical and production properties of thick-walled sheet for manufacturing gas pipelines of considerable diameter,” *Metallurg*, No. 6, 49–54 (2005).
4. Yu. D. Morozov, S. Yu. Nastich, M. Yu. Matrosov, and O. N. Chevskaya, “Provision of improved set of properties for rolled pipe of considerable diameter based on forming a steel ferrite-bainite microstructure,” *Nauk. Tekhn. Gaz. Prom.*, No. 1, 41–46 (2009).
5. I. Yu. Pyshmintsev, A. N. Boryakova, and M. A. Smirnov, “Effect of cooling rate on structure and properties of low-carbon pipe steel,” *Metallurg*, No. 8, 48–51 (2008).
6. M. V. Malakhov, G. D. Motovilina, E. I. Khlusova, and A. A. Kazakov, “Structural inhomogeneity and methods of reducing it in order to improve structural steel quality,” *Vopr. Material.*, No. 3(59), 52–64 (2009).
7. V. M. Schastlivtsev, T. I. Tabatchikova, I. L. Yakovleva, et al., “Features of the structure of bainite in low-carbon weldable steels after thermomechanical treatment,” *Vopr. Material.*, No. 3(59), 26–38 (2009).