PIPE STEELS

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A STUDY OF THE MICROSTRUCTURE OF NIOBIUM-MICROALLOYED PIPE STEEL AFTER DIFFERENT MODES OF CONTROLLED ROLLING WITH ACCELERATED COOLING

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The phase composition and mechanical properties of rolled thick sheets from pipe steel 05G1MB produced by different modes of controlled rolling with accelerated cooling are studied. The effects of the temperature and deformation regime, of the final rolling temperature, and of the temperature of the start of accelerated cooling on the phase composition of the rolled sheets are considered. The temperature range in which intense reduction during rolling should be avoided in order to keep niobium in the solid solution of the γ-phase and to ensure subsequent segregation of fine niobium carbides in ferrite is determined.

INTRODUCTION

Today's Russia new fields are developed and new pipelines driven, which increases considerably the demand for rolled sheets for pipes with a high strength category (X80 in accordance with API-5L and higher) and enhanced cold resistance. Ferrite-pearlite steels do not ensure the required mechanical properties and cold resistance. In this connection it is necessary to produce rolled sheets for pipes from steels of the ferrite-bainite class, which can match the demanded level of properties. The most economically effective process for producing such sheets is controlled rolling with accelerated cooling (CR + AC). The process of accelerated cooling is based on controlling the transformation of austenite after the deformation has been finished. The main parameters of the process are the temperature at which the cooling is begun (t_{ci}), the temperature at which the accelerated cooling is interrupted (t_{cf}), and the rate of the cooling (vcool) [1]. Changing the parameters of CR and AC we can obtain different structural components in a sheet, i.e., ferrite-pearlite, ferrite-bainite, ferrite-bainite with regions of martensite, and a structure of polygonal ferrite in combination with acicular ferrite.

As a result of the use of accelerated cooling the fineness of the products of the intermediate transformation increases, the type and the proportion of the structural components changes, the efficiency of the precipitation hardening increases due to refinement of the particles of the carbonitride phases, the dislocation density increases, and the banding is removed (due to deceleration of the diffusion of carbon). Thus, the use of accelerated cooling ensures substantial strengthening of the metal without additional alloying.

The aim of the present work consisted in studying the structure and mechanical properties of microalloyed pipe steel 05G1MB after different modes of controlled rolling with accelerated cooling.

METHODS OF STUDY

We studied test pieces cut from four sheets 12 mm thick fabricated from a test converter heat of steel of type 05G1MB (0.06% C, 0.2% Si, 1.44% Mn, 0.008% P, 0.003% S, 0.1% Ni, 0.15% Cu, 0.016% Ti, 0.029% Al, 0.016% Mo, 0.051% Nb, 0.005% N, $C_e = 0.63$, $P_r = 0.16$). The sheets were rolled in a 5000 LPTs-3 mill of the Severstal' Company from continuously cast slabs.

The carbon equivalent was determined by the formula

$$C_e = C + Mn/6 + (Cr + Mo + V)/5 + (Ni + Cu)/15.$$

The parameter of crack resistance in welding was determined by the formula

$$P_r = C + V/10 + Mo/15 + Cr/20 + Mn/20 + Cu/20 + Si/30 + Ni/60 + 5B.$$

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Before rolling, the slabs with a thickness of about 200 mm were heated to 1170°C. The rolling was performed in a controlled mode in two stages. Sheets *3* and *4* were rolled with long-term interim cooling between roughing and finishing in contrast to sheets *1* and *2* that were rolled virtually without a pause between the rolling stages (Table 1). The accelerated cooling after the rolling was performed in an installation for controlled cooling [1] at a rate of 30 – 40 K/sec. We recorded the temperature and strain parameters of the roughing and finishing rolling stages and the initial and final temperatures of the accelerated cooling (Table 1).

The phase transformations in continuous cooling of hot-strained austenite were studied with the help of a BAHR-805 dilatometer. Cylindrical test pieces ∅ 5 × 10 mm in size were heated to the temperature of austenization (1170°C) at a rate of 2 K/sec, held for 3 min, and deformed by compression in the alundum dies of the deforming attachment of the dilatometer; the mobile die was moved at a speed of 10 mm/sec. The temperature regime of the heating and of the deformation (the test pieces were subjected to tree-stage deformation with relative reduction of 20% in each stage at 1000, 880, and 850°C) was chosen so as to bring it as close as possible to the actual regime of high-temperature controlled rolling in which the deformation is finished at a point somewhat higher than Ar_3 (in steels of the type studied it ranges between 750 and 800°C).

The test sheets were used to take samples that were subjected to static tensile tests in the transverse direction (flat specimens with functional part 2″ (50.8 mm) long, impact bending tests (specimens with a sharp notch), and tests by a falling weight (full-thickness transverse specimens) at – 20°C.

The structure of the steel was studied by the methods of light microscopy, transmission electron microscopy (TEM) (JEM 200CX microscope at accelerating voltage of 120 kV; the specimens were prepared by a standard technique using electrolytic polishing), scanning electron microscopy (SEM) (the same microscope) with the use of at attachment for diffraction microanalysis (DMA), and x-ray diffractometry (XRD) ("Geigerflex" diffractometer, monochromatic copper K_β radiation, monochromator on reflected beam). We determined the morphology and the defects of the structure of the ferrite (TEM, XRD), the phase composition of the products of the transformations (TEM), and the composition and the crystal geometry parameters of the carbonitride phases (TEM, SEM, DMA).

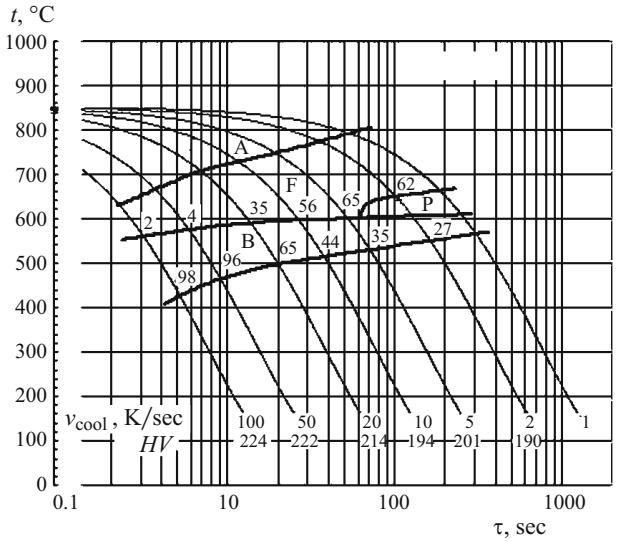

Fig. 1. Thermokinetic diagram of steel 05G1MB.

RESULTS

The thermokinetic diagram (TKD) plotted from the experimental data for steel 05G1MB is presented in Fig. 1. The TKD was used for determining the initial temperature of the ferritic transformation in the steel [for air cooling (about 1 – 2 K/sec) it was 770 – 780°C] and the final temperature of the bainitic transformation (about 600°C). In accordance with the TKD cooling of the steel from the γ-range in air yields a ferrite-pearlite structure with a low fraction of bainite; accelerated cooling (at a rate of from 5 to 50 K/sec) results in formation of a homogeneous ferrite-bainite structure.

The results of the mechanical tests showed the following: the yield strength of the four studied sheets treated by the mode of controlled rolling with accelerated cooling from various temperatures σ_y = 508 – 620 MPa, the ultimate rupture strength σ_r = 584 – 685 MPa, and the elongation δ = 29 – 31% (Fig. 2). The impact energy at – 20°C was 266 – 300 J; in the falling weight test the proportion of fibers in fracture of any specimen was 100%. For comparison, the properties of a sheet rolled from the metal of the same heat with final rolling temperature of 790°C and subsequent air cooling were as follows: σ_y = 490 MPa, σ_r = 550 MPa, and δ = 36%.

The microstructure of the four studied sheets is presented in Fig. 3. The structure of the sheets corresponds to the re-

TABLE 1. Regimes of Rolling and Accelerated Cooling of Test Sheets from Steel 05G1MB

Sheet	Initial temperature of roughing, °C	Time of interim cooling, sec	Initial temperature of finishing, °C	Final temperature of finishing, °C	Number of passes (total)	Initial temperature of accelerated cooling, °C	Final temperature of accelerated cooling, °C
1	1050	20	1025	825	20	800	575
2	970	20	970	865	19	835	565
3	985	235	930	790	23	770	525
4	995	300	860	760	19	750	540

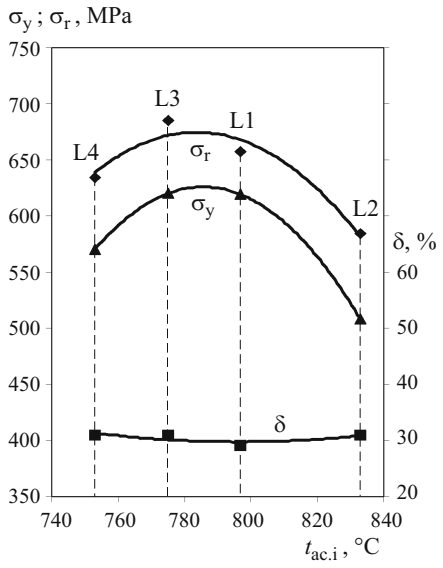

Fig. 2. Mechanical properties of four sheets (L1, L2, L3, L4) 12 mm thick from steel 05G1MB as a function of the initial temperature of accelerated cooling.

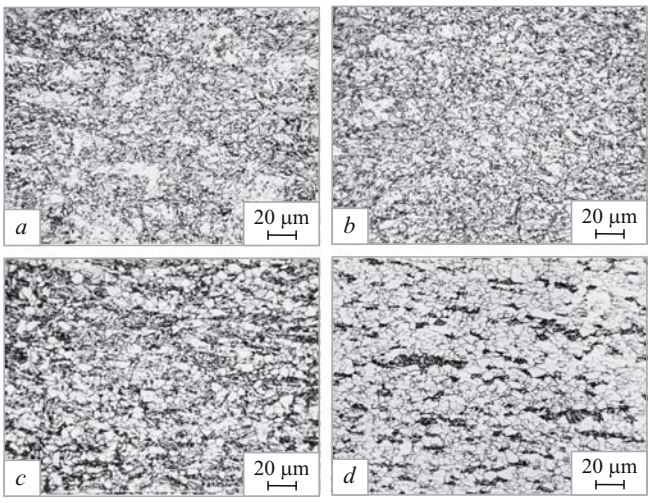

Fig. 3. Microstructure of sheets *1* (*a*), *2* (*b*), *3* (*c*), and *4* (*d*), × 200.

sults of the dilatometric study. Sheets *1* and *2* have a homogeneous fine disperse structure that virtually fully consists of products of the intermediate transformation (acicular ferrite and, possibly, a low amount of upper bainite). The structure of sheet *3* consists of polygonal ferrite (about 40%) and products of intermediate transformation (about 20% acicular ferrite and 40% low-carbon bainite). The structure of sheet 4 consists of a matrix of fine-grained polygonal ferrite with regions of high-carbon bainite (about 20%) with a tendency to formation of bainite regions in the form of bands extended along the rolling direction.

Figure 4 presents the results of a study of the tested sheets by the method of TEM. A region about 6 μm in size with a structure of acicular ferrite and a high dislocation density (about 9×10^9 cm^{-2}) can be seen in Fig. 4*a*. A similar structure has been observed in sheets *1* and *2*. Mechanically hardened polygonal ferrite with a lower dislocation density [about $(5 – 6) \times 10^9$ cm^{-2}) has the form of large regions in specimens of sheet *4* (Fig. 4*b*). Individual regions with structure of the type of upper bainite were observed in specimens of all the sheets (Fig. 4*c*). In a specimen of sheet *1* we detected regions of retained austenite with martensite or lower bainite (Fig. 4*d*). It is difficult to evaluate the fraction of the martensite-austenite (M/A) phase by the data of TEM; we can only note that such regions are encountered quite rarely.

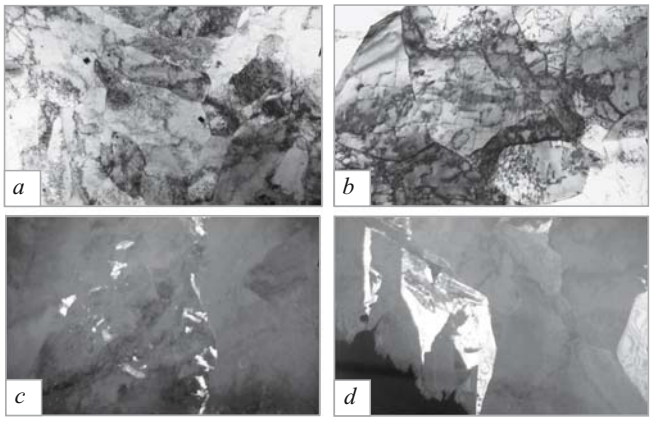

Fig. 4. Microstructure of sheets from alloy 05G1MB (TEM): *a*) region with acicular ferrite, sheet *1*, × 10,000; *b*) polygonal ferrite, sheet *4*, × 20,000; *c*) bainite, sheet *3*, × 20,000; *d*) martensite + austenite (retained), sheet *1*, × 20,000; *a*, *b*) in a light field; *c*, *d*) in a dark field.

According to the data of XRD, decrease in the final rolling temperature is accompanied by monotonic decrease in the value of microdistortions of the crystal lattice ($\Delta d/d$) from 1.6×10^{-3} in a specimen from sheet *2* to 0.4×10^{-3} in a specimen of sheet *4*. Given that the microdistortions are connected with the dislocation structure, these dependences agree with the growth in the fraction of low-dislocation polygonal ferrite upon decrease in the final rolling temperature and, accordingly, with the temperature of the start of accelerated cooling (Fig. 5).

The data of TEM and SEM show the presence of particles 0.15 – 0.6 μm in size in specimens of all the four sheets. The particles are not polishable and therefore are well detectable by SEM. The lattice parameter of these particles is not determinable by TEM because they do not transmit light. In sheet *2* we detected the finest particles of this type, which were uniformly distributed over the area of the specimen (Fig. 6*b*) and had a size of 0.15 – 0.2 μm. In accordance with the data of XRMA the particles contain 33 – 25 at.% Nb and Ti and form complex carbonitrides, i.e., (Ti, Mn)CN. Some particles contain only Nb (NbCN carbonitride).

In specimens of sheets *1*, *3*, and *4* the particles of (Ti, Nb)CN are coarser (0.3 – 0.6 μm) but their content is

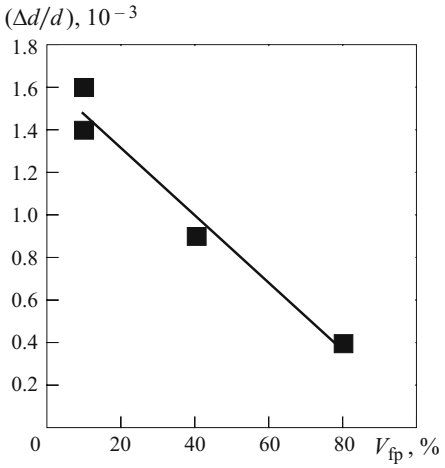

Fig. 5. Averaged size of microdistortions of crystal lattice as a function of the volume fraction of polygonal ferrite V_{fp} (data of XRD).

lower than in the specimen of sheet *2* (Fig. 6*a*). The content of Nb in these particles is less than about 15 – 20 at.%. Particles of this type form by the mechanism of epitaxial segregation of (Ti, Nb)C on the earlier formed TiN particles [2].

Relatively small particles 0.04 – 0.1 μm in size, which transmitted light in the TEM study, were detected only in sheet *2*. The microdiffraction patterns show that these particles have an fcc lattice with parameter of 0.422 – 0.424 nm, which corresponds to a TiN compound [2]. The possibility of microdiffraction analysis of these particles is connected with their small size (in contrast to the particles of the first type), but this complicates their observation and analysis of their composition by SEM. No crystal geometry tie exists between the lattices of the fcc particles and of the bcc ferrite; one ferrite grain can contain particles with arbitrary orientation.

In addition to coarse particles specimens of sheets *1*, *3*, and *4* contain dispersed particles of NbC at most 4 – 5 nm in size (Fig. 7). The images of the particles were obtained in weak smeared reflections of type 200 of an fcc lattice with parameter about 0.44 nm, which corresponds to a NbC compound. The particles are regularly oriented with respect to the ferrite lattice, i.e., $(110_{NbC} \parallel 100_F)$, $(001_{NbC} \parallel 001_F)$. The actual number of NbC particles in the volume is greater than can be detected in dark-field images, because one ferrite grain contains at least two possible orientations. The particles of NbC about 5 nm in size are noncoherent to the matrix due to their equiaxial shape and due to generation on defects of the crystal structure of the matrix, i.e., on subgrain boundaries and dislocations (Fig. 7*a* and *c*). The finer particles shaped as thin plates (Fig. 7*b*) can be partially coherent [3].

We have not detected such particles in the specimen of sheet *2*. Their absence was confirmed by the absence of NbC reflections on the microdiffraction pattern from plane $(001)^*_\alpha$ of the reciprocal lattice of ferrite.

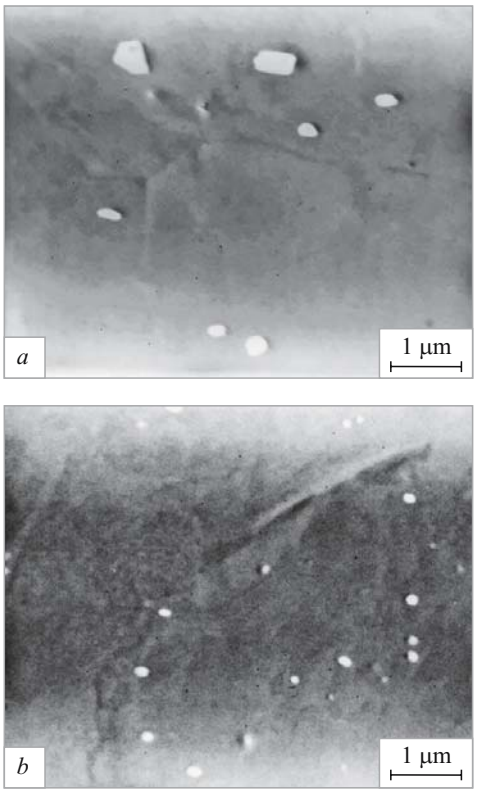

Fig. 6. Niobium and titanium carbonitrides (Nb, Ti)(CN) segregated in austenite (SEM): *a*) sheet *1* (a region where the number of particles is greater than in the other checked regions); *b*) sheet *2*.

DISCUSSION

Generalized results of the study of the phase composition of the rolled sheets, of the carbide and carbonitride phases, and of the defects of the crystal structure are presented in Table 2.

The data obtained allow us to explain the difference in the strength characteristics of the four sheets.

Sheet *1* has been cooled from the austenite range and has quite high strength characteristics ensured by the structure of acicular ferrite with elevated dislocation density; it is additionally reinforced with bainite and a certain amount of an M/A phase. Disperse particles of NbC make an additional contribution to the strengthening of the ferrite [4].

Sheet *2* has also been cooled from the austenite range and its structure consists predominantly of acicular ferrite and a low fraction of upper bainite; however, the absence of precipitation hardening by NbC results in the lowest level of strength characteristics (see Fig. 2).

Sheet *3* has been cooled from the upper part of the $(\gamma + \alpha)$ range and possesses the highest strength; its structure consists of bainite (40%), acicular ferrite (20%), and polygonal ferrite (40%) reinforced with particles of NbC.

Accelerated cooling of sheet *4* has been started from the middle part of the $\gamma + \alpha$ range and therefore the structure is

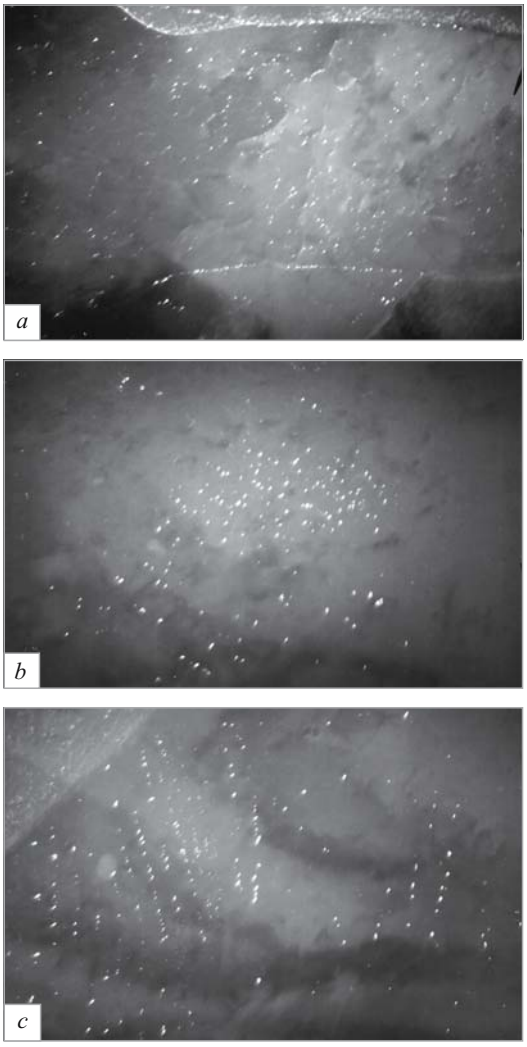

Fig. 7. Fine particles of NbC segregated in ferrite (TEM, dark field), × 50,000: *a*) sheet *1*; *b*) sheet *3*, *c*) sheet *4*.

primarily represented by polygonal ferrite (reinforced by mechanical hardening during rolling and by segregation of NbC particles on dislocations) and carbon-bearing bainite (about 20%). The strength of this sheet is lower than that of sheet *3* because the amount of the strong acicular transformation products has been decreased.

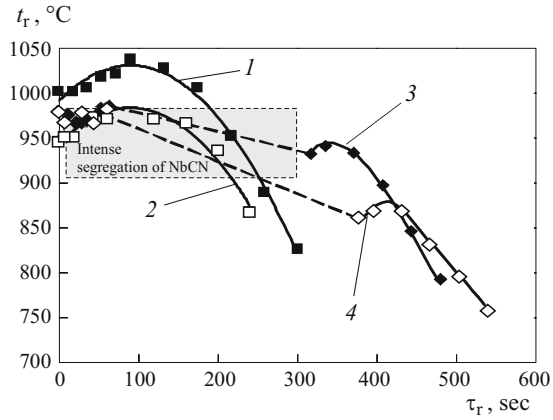

Fig. 8. "Temperature – rolling time" diagram for tested sheets *1*, *2*, *3*, and *4* from steel 05G1MB: dashed lines) interim cooling; solid lines) deformation.

In order to determine the cause of formation of (Ti, Nb)CN carbonitride particles of various sizes and of the absence of disperse NbC particles we compared the results of the electron-microscope study and the temperature and deformation mode of production of the sheets.

In accordance with published data [2] the most intense segregation of NbCN in the austenite of low-alloy C – Mn – Nb steels under deformation occurs in a temperature range of 970 – 900°C. The time interval is also restricted, i.e., 10 sec after the moment of the start of the deformation the segregation of particles begins at a high rate, which is connected with accumulation of defects in the austenite and with growth in the number of possible nucleation sites of the particles after which the segregation of NbCN decelerates; in approximately 300 sec the segregation process stops.

The temperature and deformation regimes of the production of test sheets differed noticeably (Fig. 8). In sheet *1* the major part straining (to $\delta = 91.5\%$) occurred at relatively high temperatures ($t_{def} = 1050 – 1000$°C). A considerable amount of niobium remained in the solid solution of austenite and segregated in the form of fine particles of NbC in ferrite on dislocations and over grain and subgrain boundaries during the $\gamma \rightarrow \alpha$ transformation (see Fig. 7*a*). The presence of high amount of niobium in the solid solution before the cooling was started could raise the stability of the austenite

TABLE 2. Parameters of the Structure of Rolled Sheets from Steel 05G1MB

Sheet	Structural components, %				Particle size, μm			Averaged value of microdistortions, $(\Delta d/d)$, 10^{-3}
	AF	PF	B	M/A	(Ti, Nb)(CN)	TiN	NbC	
1	80	10	10	+	0.3 – 0.5	–	≤ 0.005	1.4
2	80	10	10	–	0.15 – 0.2	0.04 – 0.1	–	1.6
3	20	40	40	–	0.3 – 0.5	–	≤ 0.005	0.9
4	–	80	20	–	0.3 – 0.6	–	≤ 0.005	0.4

Notations: AF) acicular ferrite; PF) polygonal ferrite; B) bainite; M/A) martensite + austenite.

and cause formation of islands of an M/A phase detected in the specimen of the sheet (see Fig. 4*d*).

In sheet *2* straining to $\delta = 91.5\%$ occurred at $t_{def} =$ 970 – 930°C, i.e., in the range of intense segregation of niobium carbonitrides we observed numerous uniformly distributed relatively fine particles of NbCN and (Ti, Nb)CN (Fig. 6*b*). We detected "pure" TiN particles without features of epitaxial segregation of Nb on them only in the specimen of this sheet, because the segregation of NbCN particles in this case did not require a heterogeneous kind of formation. It can be assumed that virtually all of the niobium has segregated in austenite, which explains the absence of segregation of fine NbC particles in ferrite.

The regime of rolling of sheets *3* and *4* ensured lower total strain at the temperatures of intense segregation of NbC and involved interim cooling between the roughing and the finishing. The cooling (in the absence of deformation) increased the rolling time, which made it possible to avoid straining in the range of intense segregation of carbonitrides and to preserve a large enough part of niobium in the solid solution of austenite; after the $\gamma \rightarrow \alpha$ transformation it segregated in ferrite in the form of nanoparticles of NbC.

CONCLUSIONS

1. Depending on the temperature and deformation regime of controlled rolling in combination with accelerated cooling we have obtained a wide spectrum of structures in steel 05G1MB, i.e., polygonal and acicular ferrite, upper bainite, an M/A phase, and particles of carbonitrides of different morphology.

2. The mechanical properties of the rolled sheets in this case vary in a wide range. The highest strength characteristics are attained when the process yields a disperse microstructure consisting of fine-grained polygonal and acicular ferrite and bainite. This is achieved when the process involves cooling from the top part of the $\gamma + \alpha$ range to a temperature of 550 – 500°C.

3. Correct choice of the temperature and deformation regime of controlled rolling makes it possible to control the content of niobium in the solid solution and ensure segregation of fine particles of NbC in ferrite, which makes a substantial contribution to the strengthening of the metal. Approximate evaluation of the contribution of precipitation hardening in the given steel gives 100 MPa at an optimum morphology of the particles.

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