Study of Methods for Increasing Ductility and Formability of Cold-Rolled Ti-Stabilized IF Steels

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Abstract. Study of the influence of the chemical composition and parameters of the complex treatment technology on characteristics of the microstructure, phase precipitates, the solid solution state, and mechanical properties was carried out for hot-rolled and cold-rolled Ti-stabilized IF steels. Cold-rolled products were annealed according to the regime of continuous hot-dip galvanizing units. The following methods of investigation were used: light, scanning and transmission electron microscopy, mechanical properties testing, thermodynamic analysis of phase stability conditions, and determination of the interstitial impurities content in a solid solution by internal friction. The possibility and conditions of a significant increase in ductility, formability of cold-rolled steels are established. They are obtained not by the traditional way consisting in reducing the content of interstitial elements and impurities, but by giving them a favorable form of existence. This is achieved by optimizing the chemical composition, first of all, the sulfur content, and parameters of thermo-deformation treatment of steel, which ensure complete carbon binding in TiC_2S_2 as a result of TiS transformation already at the hot-rolling stage. To reduce the yield and tensile strength, it is necessary to decrease the content of interstitial elements and to increase the temperature of recrystallization annealing of cold-rolled steel.

1. Introduction
The rapid development of the automotive industry, high-speed transport, instrumentation, and industrial, household, and other technical objects makes it necessary to produce elements and assemblies of an increasingly complex shape by progressive stamping methods. For these purposes, cold-rolled steels, including coated ultralow carbon IF steels, are widely used [1,2]. However, they, as a rule, have insufficiently high indicators of ductility and formability (relative elongation of 44–50%, coefficients of normal plastic anisotropy, \( r \approx 2.0–2.2 \), and strain hardening, \( n \approx 0.20–0.24 \)), which makes it difficult or impossible to obtain stamped products of complex shape. Cracks and other defects are observed on the body of the metal that hinder the improvement of the design and service characteristics of new technical means. In world practice, an increase in ductility and formability of rolled products is achieved mainly owing to the use of a high-purity metal charge and technological methods for obtaining low (less than 0.002%) carbon and nitrogen contents in IF steels [3–6]. This is associated with significant costs. On the other hand, it is well known that impurities and interstitial elements participate in the formation of a number of phase precipitates in IF steels, including complex composition [7–16], and not their absolute content is important, but the form of the presence of impurities and interstitial elements [13–17]. Therefore, a considerable problem is to establish the possibilities and conditions for achieving fundamentally improved indicators of ductility and formability of cold-rolled steels using not the...
traditional way of obtaining an extremely high degree of impurity and interstitial element cleanliness, but by giving them a favorable form of existence. This is the subject of present study.

2. Materials and methods
The most economical cold-rolled Ti-microalloyed (stabilized) IF steels annealed in continuous units were studied. Attention was focused on continuous hot-dip galvanizing unit, CHGU, because obtaining high ductility and formability in unit of this type is difficult due to the limited duration of stay of the strip in it. The steels were smelted in a vacuum induction furnace with a magnesite crucible with a steel capacity of 7–8 kg and casted in a vacuum in one ingot. The results of determining their chemical composition are presented in table 1.

Table 1. The chemical composition of the studied steels.

| Melting no. | Content of elements, wt. % |
|-------------|----------------------------|
|             | C  | Si  | Mn  | P  | S  | Cr | Ni  | Cu  | Al  | N  | Mo | V  | Nb | Ti |
| 1           | 0.004 | 0.01 | 0.10 | 0.007 | 0.007 | 0.02 | 0.02 | 0.03 | 0.041 | 0.003 | 0.003 | 0.001 | - | 0.057 |
| 2           | 0.006 | 0.01 | 0.11 | 0.796 | 0.067 | 0.03 | 0.02 | 0.02 | 0.042 | 0.005 | 0.004 | 0.002 | - | 0.060 |
| 3           | 0.004 | 0.01 | 0.11 | 0.782 | 0.286 | 0.02 | 0.02 | 0.03 | 0.036 | 0.004 | 0.002 | 0.002 | 0.001 | 0.059 |
| 4           | 0.003 | 0.01 | 0.09 | 0.006 | 0.007 | 0.03 | 0.01 | 0.02 | 0.035 | 0.004 | 0.001 | 0.001 | - | 0.063 |

The ingots were heated to 1220 °C, held for 1 hour, and rolled on DUO-300 reversible hot rolling laboratory mill with 12–14% reduction in the last pass into strips of 3 mm thickness. After rolling end at various finishing temperatures, Tf, the strip was cooled in a stream of air with a cooling rate of 10–15 °C/s to the temperature of the strip coiling into a roll, Tc. Then it was placed in a furnace heated to this temperature, held for 30 minutes, followed by cooling with the furnace, which simulated the cooling of a roll. The main parameters of the thermo-deformation metal treatment are presented in table 2.

Table 2. Parameters of the thermo-deformation treatment of steels.

| Melting no. | Tf, °C | Tc, °C | Cooling rate to Tc, °C/s |
|-------------|--------|--------|-------------------------|
| 1           | 885    | 740    | 10-12                   |
| 2           | 920    | 720    | 13-15                   |
| 3           | 930    | 720    | 13-15                   |
| 4           | 920    | 720    | 13-15                   |

Subsequently, hot-rolled strips were pickled and rolled at the DUO-QUARTO 320 cold rolling laboratory mill with 75% reduction into strips of 0.75 mm thick. The obtained cold-rolled steel was subjected to recrystallization annealing on a universal testing machine according to a regime simulating annealing in the CHGU with a strip moving rate of 100 m/min. The main parameters were as follows: annealing temperature, Ta – 810–830 °C (see below), delayed cooling to 690 °C, accelerated cooling to 475 °C, accelerated cooling after a galvanizing bath to 190–200 °C. After annealing, the bands were subjected to temper rolling with a relative elongation of 0.8%. Samples were made of the obtained hot-rolled and cold-rolled products to study the characteristics of the structural state and the properties of the metal.

The microstructure was studied by methods of light microscopy, scanning electron microscopy, SEM, using a JSM-6610LV (JEOL) instrument equipped with an INCA Energy Feature XT energy dispersive microanalysis system, INCA Wave 500 wave dispersion spectrometer, and transmission electron microscopy, TEM, using a JEM200CX microscope. Herewith, the type of nanosized precipitates was determined by the original method based on the analysis of their reflections in
microdiffraction images [18]. The mechanical properties and carbon content of the solid solution were determined by tensile methods on a HECKERT FP-100/1 tensile testing machine and internal friction, respectively. The thermodynamic calculation of the regions of existence of phases in the steel under study was carried out using the approaches presented in detail in [19, 20].

3. Results
By means of light microscopy the average grain diameter was measured in the rolling direction (dx) and in the direction perpendicular to the rolling direction (dy). Using these values the average grain diameter (da) was calculated as the arithmetic mean of dx and dy, as well as the ratio dx/dy, which is in fact, a characteristic of the degree of elongation of the grain shape. The coefficient of variation characterizing the heterogeneity of the grain structure was found as the ratio of the standard deviation to the average grain size. The results of the study of the microstructure and mechanical properties of hot-rolled products are presented in tables 3.

| Melting No. | dx, mkm | dy, mkm | d/dy | Coefficient of variation | σ0.2, MPa | σB, MPa | δ, % |
|-------------|---------|---------|------|-------------------------|-----------|---------|------|
| 1           | 15.2    | 16.2    | 1.07 | 0.59                    | 201       | 291     | 43.4 |
| 2           | 14.2    | 16.2    | 1.14 | 0.52                    | 163       | 289     | 43.2 |
| 3           | 14.3    | 16.3    | 1.14 | 0.55                    | 199       | 287     | 43.1 |
| 4           | 14.2    | 16.0    | 1.13 | 0.50                    | 159       | 283     | 44.5 |

The test results of the mechanical properties and parameters of annealing of cold rolled steels studied are presented in table 4.

| Melting No. | T_a, °C | σ0.2, MPa | σB, MPa | δ, % | r  | n   |
|-------------|---------|-----------|---------|------|----|-----|
| 1           | 825     | 143       | 294     | 48.7 | 2.05 | 0.27 |
| 2           | 830     | 148       | 313     | 50.9 | 2.09 | 0.26 |
| 3           | 810     | 158       | 312     | 46.9 | 2.06 | 0.26 |
| 4           | 830     | 128       | 279     | 51.7 | 2.40 | 0.26 |

The results of thermodynamic calculation showed that the main types of phase precipitates in the studied steels are TiN, TiS, Ti₄C₂S₂, TiC. Nitrogen at high temperatures is completely bound in TiN. Depending on the chemical composition and processing parameters of the steel, carbon is predominantly involved in the formation of Ti₄C₂S₂ or TiC. This was confirmed by the TEM study, which found out a significant amount of such precipitates in rolled metal (figure 1).

**Figure 1.** Typical view of precipitates in cold rolled steel: a - titanium carbosulfide (melting No. 2, x20000), b - titanium carbide (melting no. 1, x30000).
4. Discussion

Data of table 3 show that a slightly larger grain size was obtained for hot-rolled steel of melting No.1 produced using a lower finishing temperature of rolling and a higher temperature for coiling the strip into a roll. This may be due to a lower cooling rate of the strip after the rolling end. For rolled steels of melting No. 2, 4 the values of yield strength are lower (table 3). Determination of the carbon content in the solid solution by the method of internal friction showed that the samples No. 1 and 3 contain about 6 and 7 ppm carbon, respectively, while the carbon in the solid solution in steels No. 2 and 4 is absent. The calculation according to the generally accepted ratio of the solid solution hardening of steel caused by the presence of 6-7 ppm carbon in the solid solution showed that this value is about 25-30 MPa. Thus, the increased values of the yield strength of rolled steels of melting No. 1 and 3 are mainly associated with solid solution hardening with carbon. Therefore, the preservation of even minor carbon concentrations in a solid solution can lead to a significant increase in the yield strength.

From the data of table 4 it can be seen that the highest ductility and formability are obtained for cold-rolled steels No. 2 and 4, which do not contain carbon in a solid solution of hot-rolled products. According to the results of the study by SEM, TEM, and local X-ray spectral analysis of rolled products it was shown that this is achieved due to the preferential binding of carbon in TiC2S2 (figure 1a). There are much more such precipitates in rolled products from steel of melting No. 4 and, especially No. 2, than in the case of melting No.1 and 3. Analysis of the phase stability conditions in the studied steels confirmed the conclusion made. It should be noted that steels of melting No. 2 and 4 are characterized by the highest and the lowest carbon content, respectively, which determines the amount of TiC2S2 precipitates formed.

The kinetics studies of strain-induced TiC2S2 precipitation performed for IF steel of model composition close to investigated, (wt.%): 0.004C, 0.02Si, 0.18Mn, 0.006P, 0.014S, 0.09Ti, 0.05Al, 0.004N [12], led to the following conclusions. The “nose” of the C-shaped curve, corresponding to the external combination of thermodynamic and kinetic stimuli for the formation of precipitates, is at a temperature of about 940 °C. If TiS precipitates are present in the steel, the formation of TiC2S2 precipitates occurs at 910–950 °C by heterogeneous nucleation on the surface and transformation of TiS to TiC2S2. This process is rather fast and ends in 20-30 s. Thus, there are all prerequisites for the complete binding of carbon from a solid solution to TiC2S2 during hot rolling of steel and its end at 910–950 °C. Studies performed for steels with a low sulfur content up to 0.004 - 0.005 wt.% showed that in the absence or low TiS content, the formation of TiC2S2 precipitates occurs through self-nucleation and growth of nuclei of this phase. This leads to a significant increase in the time of complete transformation, reaching values of more than 1000 s. As a result, predominant carbon binding occurs not in TiC2S2, but in TiC and can create the preconditions for its conservation in the solid solution. This is actually observed in the case of steels No. 1 and 3.

The noted features are also confirmed by the results of the thermodynamic calculation of phase stability conditions. In particular, they indicate that upon heating billets of steel of melting No. 1, 2, and 4 for rolling to 1220 °C, titanium sulfide is formed in them. This, during subsequent hot rolling, facilitates the formation of titanium carbosulfide precipitates by transforming TiS precipitates already present and leads to more efficient carbon removal from the solid solution. Moreover, due to the higher finishing temperature of rolling and the mobility of atoms of the phase-forming components, the formation of titanium carbosulfide precipitates in steels No. 2 and 4 is more intense and leads to the complete removal of carbon from the solid solution. To a lesser extent, the transformation under consideration in steel of melting No. 1 leads to a decrease in the fraction of carbon bound to titanium carbosulfide, to incomplete subsequent binding to titanium carbide and to its conservation in solid solution.

Due to the low sulfur content in steel of melting No. 3, during the heating of billets for rolling, TiS formation does not occur at all. Therefore, the precipitation of titanium carbosulfide occurs by the mechanism of independent nucleation and the growth of precipitates of this phase, which has a significantly lower rate compared to the transformation of TiS precipitates. As a result, a significant part of the carbon is bound into finely dispersed titanium carbide precipitates and remains in solid solution.
The aforementioned circumstances lead to lower ductility and formability of cold-rolled steel of No. 1 and, especially, No. 3, compared to melting No. 2.

For cold-rolled products from all the studied steels, the uniform microstructure and rather high ductility and formability indicators were obtained. Nevertheless, for steels No. 2 and 4 they are higher than in the case of steels No. 1 and 3. This is due to the predominant bonding of carbon from the solid solution to TiC5S2 already at the stage of hot rolling of steel. It is important to note that the content of impurities and interstitial elements in steels No. 2 and 4 is at a level no lower than in steels No. 1 and 3, which indicates the possibility of a significant increase in ductility and formability by giving them a favorable form of existence. Analysis of the data in table 4 shows that to obtain low values of strength characteristics, it is necessary to reduce the content of interstitial elements. An increase in the temperature of recrystallization annealing of rolled products also contributes to this.

5. Conclusions
Thus, the results obtained unambiguously indicate the possibility of a significant increase in the ductility and formability of cold-rolled IF steels annealed according to the CHGU regime by giving a favourable existence form to the impurities and interstitial elements. For this, the sulfur content should be sufficiently high (at least 0.006 wt.%) to ensure the stability of TiS precipitates during heating of the steel for rolling, and the possibility of complete carbon binding in TiC5S2 already at the stage of hot rolling. This is facilitated by the use of high finishing temperatures of rolling 910-950 °C, at which TiC5S2 precipitates are formed with a significant rate due to the transformation of TiS. Otherwise, predominant carbon binding occurs not in TiC5S2, but in TiC, which can create the prerequisites for its conservation in solid solution and lowering the level of properties of rolled products. A decrease in strength characteristics is favoured by a decrease in the content of interstitial elements and an increase in the temperature of recrystallization annealing of cold-rolled steel.

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