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DEVELOPMENT OF TECHNOLOGICAL METHODS OF ADDITIONAL GRAIN REFINEMENT FOR PRODUCTION OF COLD RESISTANT Nb-BEARING PLATE STEEL WITH SMYS 450-485 MPA AND THICKNESS UP TO 40 MM

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Abstract

A continuous demand for thick plates for structural applications with yield strength of 450-485 MPa, high toughness and cold resistance requires the development of new methods of additional grain refinement at all stages of thermo-mechanical control process (TMCP). The effect of Ti-Nb precipitates morphology on grain growth during reheating was investigated. It has been shown that Nb is a key element for grain growth control. Submicron Nb carbonitrides suppress austenite grain growth at lower slab reheating temperatures. The effect of Ti additions on suppression of grain growth during low-temperature reheating is negligible. Parameters of reheating such as duration and temperature were adjusted in order to maximize the effect of Nb microalloying and to receive finer austenite grain before hot rolling. Grain refinement in the course of hot deformation was studied through physical modeling. Using the recrystallization model, new rolling schedules were introduced. Industrial trials were conducted; the effect of reheating and deformation parameters on cold resistance of steel was investigated. Implementation of the results of given study into production practice made it possible to produce plates with excellent set of properties, including strength, toughness, and cold resistance.

Introduction

The production of structural plates with high cold resistance, as well as high ductile and brittle crack propagation resistance faces several issues, in particular, provision of the required level of toughness, Z-properties, cold resistance, etc. For plates with thickness of 30 mm and above (large-diameter pipe applications), provision of consistent properties during DWTT at low temperatures is the most challenging issue. Modern technologies for production of thick plates are based on techniques that provide fine-grained structure of steel [1, 2]. Thermo-mechanical control process (TMCP) is the most cost-efficient technology for producing fine-grained thick plates. However, application of traditional two-stage process with heating up to 1170-1200ºC (and sometimes higher) does not always provide the required set of properties. The flexibility of TMCP with modern reversing hot mills allows enhancing the process so that additional contribution is made to the formation of fine-grained microstructure with high resistance to ductile and brittle failures. Main stages of the TMCP process that form the microstructure of flat steel are reheating of slabs, severe deformation performed in mill stands within specific temperature ranges, and accelerated cooling of plates. The thickness of a slab is critical for production of thick plates, since it is necessary to provide sufficient level of total deformation of both roughing and finishing stages of rolling in order to create maximum possible S_v value. However, the continuous-cast slabs suppliers often bring out some limitations. Another group of limitations is presented by the necessity to produce plates of maximum possible dimensions. In some cases it limits the deformation value per pass, which is especially important at the first stages of rolling.

In terms of obtaining fine-grained microstructure, the main task during slab reheating is to prevent excessive grain growth during the heating and homogenizing holding. It is assumed that Nb and Ti serve as the main elements that suppress grain growth during reheating for rolling in modern microalloyed steels. Nb and Ti form carbides and nitrides with low solubility which can restrain the migration of austenite grain boundaries [3]. Al nitrides and V carbides and nitrides with lower solubility temperatures make no significant contribution to grain growth suppression in case of relatively high reheating temperatures, characteristic for TMCP [4, 5].

Experimental

Research of the heating temperature and holding time influence on austenite structure of microalloyed structural steels was conducted in Vyksa Steel Works research laboratory [6]. It was determined that austenite structure of a steel containing 0.06% C, 0.21% Si, 1.8% Mn, 0.05% Nb, 0.017% Ti, 0.17% Mo with Ni, Cu, Cr additions prior to the rolling can be divided into three types depending on the heating parameters: fine-grained, coarse-grained, and mixed (Figure 1).

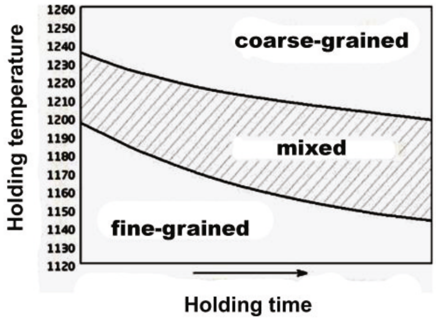


Figure 1. Structural conditions of austenite in microalloyed steels after reheating [6]

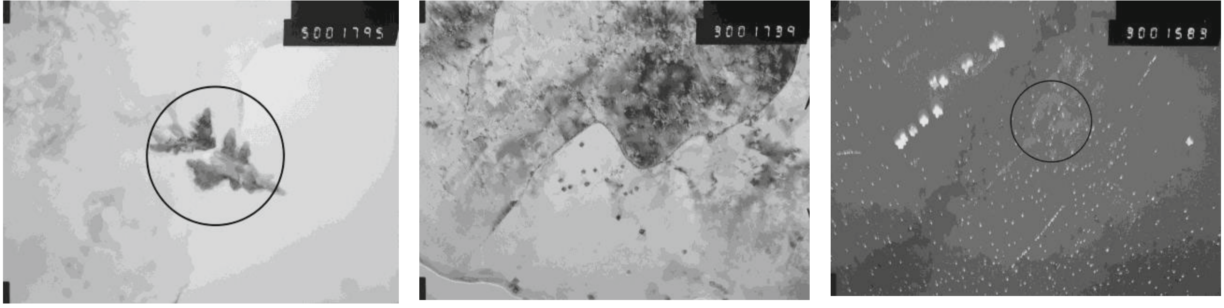
Calculation of solvus temperature of niobium carbonitrides $T_{sNb(C,N)}$ for the specified composition was made by means of Thermo-Calc software. It was found to be 1121°C. It has been found that significant coarsening of austenite grains can be attributed to the temperature range of dissolution of niobium carbonitrides. A microstructure study was conducted to investigate the causes of abnormal grain growth upon exceeding certain temperature and time parameters of the reheating process. Samples taken from industrial slabs with the specified chemical composition were heated in electric tube furnace with controlled atmosphere (argon) at the rate of 7ºC/min up to the temperatures within the range of 1160-1250ºC, held for 30 min and water quenched. The comparison of microstructure of the samples after reheating to 1160ºC, 1190ºC, and 1250ºC, and subsequent quenching has revealed significant differences both in size and distribution of constituents (Figure 2).


a B c

Figure 2. Microstructure of quenched samples after reheating to 1160°C (a), to 1190°C (b), to 1250°C (c). Etchant: 2% nitric acid

Considering that martensite packet size after quenching is governed by the size of the former austenite grain [7], it was concluded that after heating to 1160ºC the austenite structure is homogeneous and fine-grained, after heating to 1190ºC it is of mixed type, and after heating to 1250ºC it is homogeneous and coarse-grained, which complies with the results presented in Figure 1.

Transmission electron microscopy was used to study the morphology of carbonitrides in order to determine microstructural factors that influence the size of austenite grain after reheating. Morphology of carbonitrides in samples taken from as-cast industrial slabs was compared with that of carbonitrides in samples after simulated reheating. The study has shown that carbonitrides of various types can be found in as-cast slab microstructure (Figure 3). Large dendrite-like and spike-like carbonitrides, medium-sized precipitates containing Nb and Ti, and nano-sized precipitates were found. The particle size (i.e. length along main axes) ranges from several nanometers to several microns (Figures 3a, 3b). Furthermore, uniformly distributed Nb(CN) particles of 5-30 nm (third type) and areas free from such precipitates next to dendrite-like inclusions were found in as-cast slabs (Figure 3c). It appears that dendrite-like particles are being formed after slow cooling through deposition of niobium carbonitrides onto cubic-shape titanium nitride substrate with higher solidification temperature, in some cases forming chain-like clusters due to segregation phenomena. In the absence of TiN substrate, Nb(CN) particles are much finer and uniformly distributed within the matrix.



a b c

Figure 3. Dendrite-like (a), medium-sized (b), and nano-sized (TiNb)(CN) particles (c). Images produced using TEM; (a, b) bright-field image, (c) dark-field image.

Changes in morphology of carbonitrides particles after heating to 1160ºC, 1180ºC, 1250ºC and 30-min holding were studied. Both dendrite-like and nano-sized (below 10 nm) particles disappear upon heating to 1160ºC; only coarse cubic particles of TiN or 15-30 nm Nb-containing particles are observed (Figure 4a). Nb-containing particles are distributed randomly but uniformly across the area of samples, without formation of chain-like clusters.

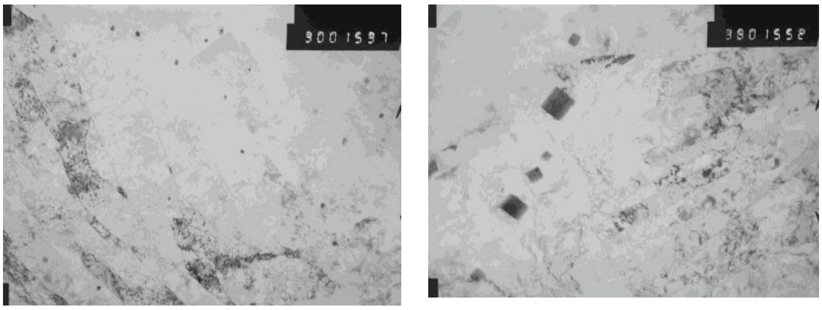

a b

Figure 4. Medium-sized carbonitrides, heating to 1160ºC, 30-min holding (a). Coarse TiN particles, reheating to 1250ºC, 30 min holding (b). TEM, bright field image.

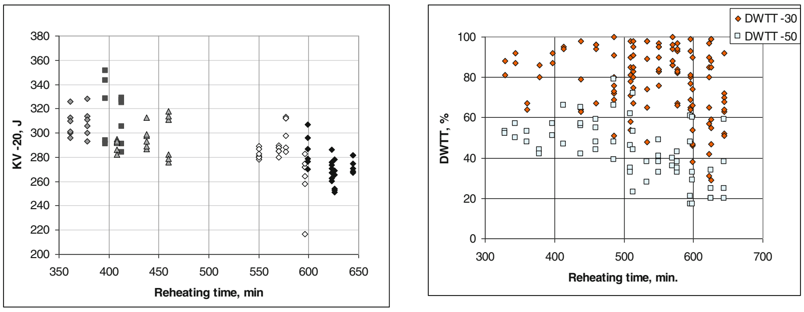
Coarse cubic particles of up to 0.3 μm (presumably TiN), sometimes forming chain-like clusters, were observed upon heating to 1180ºC (Figure 4, b). Probably, cubic particles of TiN are remnants of coarse dendrite-like precipitates [8, 9].

Discussion

After comparing the TEM data and the results of studying heating parameters versus austenite grain size, it was determined that the beginning of abnormal grain growth coincides with solution of submicron Nb carbonitrides. It was concluded that Nb is the key microalloying element that allows providing original fine-grained structure of the slab before rolling upon low-temperature reheating. It is evident that the role of titanium microalloying in formation of fine-grained microstructure is insignificant, since coarse particles of titanium nitride are located at considerable distance from each other and cannot act as a significant obstacle to the movement of boundaries. This phenomenon is especially important for plate production, since even modern high-power mills cannot provide full multiple recrystallization aimed at refining prior coarse-grained austenite structure, typical for high-temperature reheating.

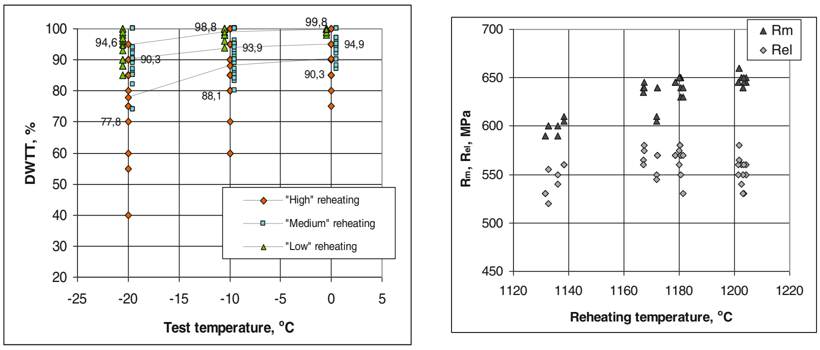
Industrial trials

Results of laboratory studies were confirmed during trial production of 40-mm plates with specified minimum yield strength (SMYS) of 450 MPa at the 5-m hot rolling mill of Vyksa Steel Works. The chemical composition of steel was 0.06% C, 0.20% Si, 1.6% Mn, 0.03% Nb, 0.016% Ti, and additions of Ni, Cu, Cr (Mo). A two-stage TMCP process with consistent parameters and various reheating modes (temperature and duration) was applied. Reheating temperatures were ranging between 1100 and 1200ºC, slabs were held in a continuous furnace within 5-12 hours. The influence of reheating parameters on absorbed Charpy energy and mechanical properties was investigated. Drop-weight tear tests (DWTT) were performed to evaluate cold resistance. Results of the industrial trials are presented in Figures 5 and 6.



a b

Figure 5. Changes of Charpy absorbed energy (KV) (a) and shear area during DWTT (b) of 450-MPa plates upon increasing the residence time of slabs in holding zone (reheating temperature 1170°C).



a **b**

Figure 6. Shear area of DWTT samples (a) and mechanical properties of 450-MPa plates (b) at different reheating temperatures.

It was confirmed that increase of reheating temperature and holding period of thick slabs has an adverse effect on Charpy absorbed energy (KV) and cold resistance (BDTT at DWTT). Furthermore, deterioration of strength properties and increase of YS/TS ratio is naturally observed upon low-temperature reheating. It should be noted that reheating temperature selection must comply with the specifications, since conventional high-temperature reheating modes (1180-1220ºC) are quite suitable to satisfy customer's requirements in most cases.

Preservation of fine-grained austenite microstructure after reheating does not exhaust the possibilities of grain refinement during TMCP. Development of optimal deformation schedules can provide additional microstructure refinement. Increase of reduction per pass does not always allow desirable refinement of austenite grain during rolling of thick plates. Furthermore, force/torque limitations of rolling mills should be considered. The important issue is uneven deformation across the slab cross-section during roughing. Outer layers are being conditioned much better than the inner ones. Furthermore, there is temperature gradient between the slab surface and inner layers. Higher temperature of the slab core promotes higher rate of grain growth after static recrystallization (SRX). Long pause between roughing and finishing also contributes to the growth of recrystallized austenite grain. In order to provide the required FRT for thick plates the finishing rolling stage is often started below Ar_3. An accelerated cooling of thick plates, especially in the middle of the cross-section, is performed at limited cooling rate, so the possibilities for refining microstructure components are limited [10]. Multi-stage rolling schedules were designed to minimize such adverse effects for production of thick plates. Custom-designed recrystallization model was used to calculate temperature and deformation parameters of rolling. Recrystallization kinetics versus temperature and deformation conditions of plate production was simulated using Gleeble instrumentation [11]. Conventional roughing schedule and roughing schedule optimized in terms of temperature and deformation parameters were simulated. Figure 7 shows microstructures of samples after rolling and quenching.

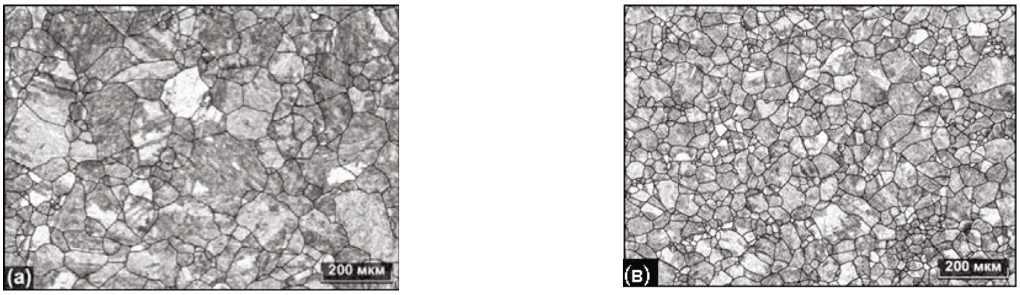
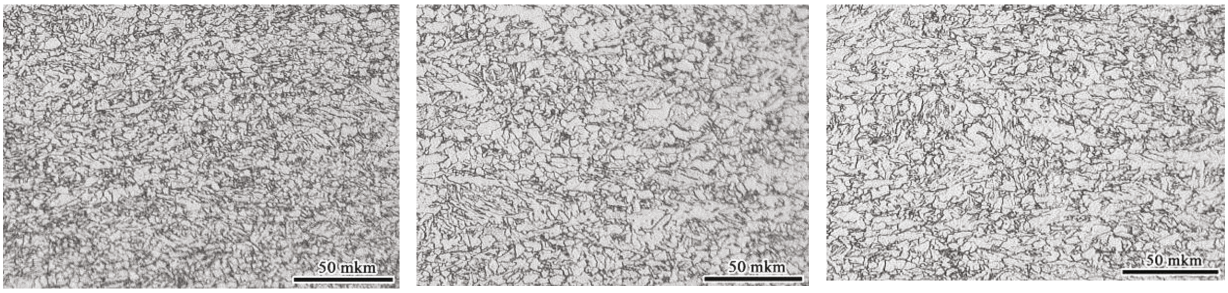


Figure 7. Austenite microstructure of samples subjected to deformation using different modes (a - conventional, b - optimized). Etchant: saturated aqueous picric acid.

It is evident that the optimized schedule allows significant additional refinement of austenite grain. The results of laboratory simulation were confirmed by industrial trials. Comparison of microstructures of 450-MPa, 40-mm plates produced using conventional (Figure 8) and optimized (Figure 9) rolling schedules demonstrated significant advantage of the developed reheating and rolling schedules in terms of obtaining fine microstructure with uniform distribution through thickness.

a **b** **c**

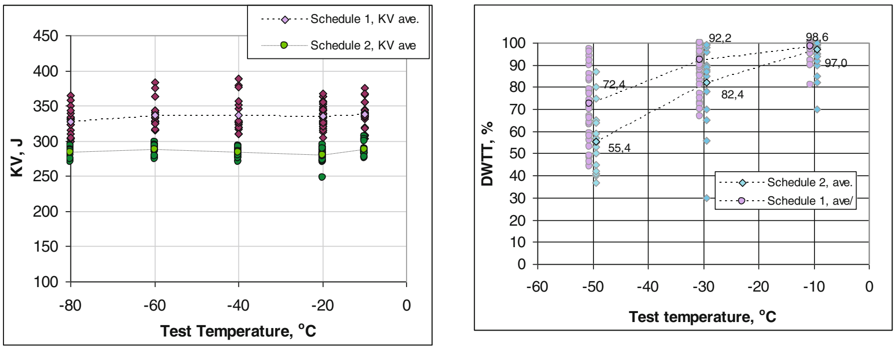
Figure 8. Microstructure of 450-MPa plate. Two-stage rolling, high-temperature reheating is applied. Etchant: 2% nitric acid.



a **b** **c**

Figure 9. Microstructure of 450-MPa plate. Multi-stage rolling using optimized schedules, low-temperature reheating. Etchant: 2% nitric acid.

Application of conventional TMCP process to thick plates (reheating up to 1180-1200ºC, two-stage rolling + accelerated cooling) results in microstructure with both non-homogeneous size distribution and type of constituents. It was shown that conventional TMCP produced banded microstructure consisting of work-hardened ferrite and coarse bainite which were found in subsurface layers (Figure 8a). The ferrite-bainite structure is sufficiently homogeneous at ¼ of the thickness (Figure 8b). However, large areas of lath-like bainite were identified in the medium plate layers due to mixed grain structure of prior austenite (Figure 8b). Optimized reheating and rolling schedules calculated via recrystallization model allowed obtaining thick plates with homogeneous microstructure both in terms of grain size distribution and type across the plate cross-section (Figure 9). The plates produced using the optimized multiple-stage process displayed better Charpy energy and shear area at DWTT than the plates produced using conventional two-stage TMCP process. The optimized TMCP process allowed reducing transition temperature T_{85} at DWTT by 15-20ºC (Figure 10). The newly-developed TMCP schedules were used for industrial production of 450-485MPa plates.



a b

Figure 10. Charpy absorbed energy (a) and shear area at DWTT (b) for the plates produced via conventional and optimized TMCP process

Conclusion

At low-temperature slab reheating undissolved Nb carbonitrides Nb(C,N) suppress abnormal austenite grain growth, which, in combination with newly-developed multiple-stage rolling schedules provides significant refinement and homogenization of microstructure of structural plates improving both toughness and cold resistance characteristics.

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