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## Physical simulation of finish rolling of microalloyed steels in isothermal conditions

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#### Abstract

The aim of this work was to establish a temperature of finish rolling stage of Nb/Ti microalloyed steel containing 0.06 wt.% C, 0.77 wt.% Mn, 0.039 wt.% Nb and 0.015 wt.% Ti, using physical simulation. Samples were subjected to laboratory simulation at a twist plastometer at high temperatures, *i.e.* between 825 and 950 °C. Five pass deformation and interpass times were selected in accordance with a processing parameters at five stand finishing hot strip mill. Restoration (recovery and/or recrystallization) behavior was evaluated by calculation of Fraction Softening (FS) and Area Softening Parameter (ASP) values. At 950 °C all individual pass stress-strain curves, FS and ASP show full recrystallization in all interpass intervals. On the other hand, with a decrease in temperature to the interval of 875-825 °C, the extent of restoration is decreasing, leading to recovery as a sole softening mechanism at 825 °C, which was confirmed by the stress-strain curve shape, and values of FS and ASP. It is assumed that, due to high supersaturation, strain-induced precipitation promoted pinning of grain and subgrain boundaries and suppressed recrystallization. Therefore, the critical temperature for finish rolling was estimated to be 825 °C.

*Keywords:* fraction softening; mechanical metallography; deformation; recrystallization, critical rolling temperatures, controlled rolling.

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### **1. INTRODUCTION**

Development of microalloyed steels is an answer to a growing demand of the modern industry for steels with improved strength, toughness and weldability [1-3]. Weight reduction in structures and low-cost manufacturing processes are constant challenges in metallurgical engineering [4]. Microalloyed steels are divided into two main groups: low and medium carbon microalloyed steels. In addition to for low carbon content values, low carbon microalloyed steels contain less than 0.15 wt.% of vanadium (V), titanium (Ti) and niobium (Nb) in total.

Addition of microalloying elements combined with adequate thermomechanical treatment (TMCP) results in significantly improved mechanical properties [3], in comparison to C-Mn steels, without expensive subsequent heat treatment. Thermomechanical treatment of low carbon microalloyed steels should provide desired shape and dimensions as well as final microstructure [5]. In that regard, hot rolling does not only provide controlling the temperature, strain ( $\epsilon$ ) and strain rate to attain the final dimension and microstructures, but it is also applied to obtain a product with desired mechanical properties [3,5].

The most common hot rolling technology, known as conventional controlled rolling (CCR) of microalloyed steels consists of rough rolling, finish rolling and controlled cooling of hot rolled steel before coiling [5-7]. Therefore subsequent heat treatment is not necessary [5]. Roughing stage is performed at temperatures that should be high enough to provide a complete static recrystallization (SRX) during the interpass time. Due to short interpass time and adiabatic heating, it can be assumed that finish rolling happens under isothermal conditions [5]. As shown in Figure 1,

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the roughing stage is performed above the non-recrystallization temperature,  $T_{nr}$ , temperature above which recrystallization is still complete [8-11]. Hot rolling at a finish rolling mill, is performed between  $T_{nr}$  and  $A_3$ , temperature below which recrystallization is suppressed as can be seen in Figure 1 [5,7-9,10,12]. Suppression of the recrystallization process depends on formation of precipitates (nitrides and/or carbonitrides) based on microalloying elements [13-17]. When additional strengthening is required, finish rolling might be performed in the dual phase temperature region (between  $A_1$  and  $A_3$ ), where ferrite grains are deformed as well as austenitic grains, which results in formation of substructure in deformed ferrite grains. Finish rolling is followed by controlled cooling or even accelerated cooling, which results in even finer grained microstructure. The non-recrystallization temperature,  $T_{nr}$  [8,9,16,18] is determined based on the Boratto test [7,16-18], while the  $T_{rl}$  (the lowest temperature for full recrystallization) and  $T_{rs}$  (the highest temperature for suppression of recrystallization) are also reported [8-11]. the non-recrystallization temperature,  $T_{nr}$ , is usually considered to be close to the  $T_{rl}$ , or temperature for 50 % of SRX, *i.e.* temperatures below which finish rolling can be performed [8-11,16,18].



Figure 1. Schematic illustration of thermomechanical processing

Therefore, determination of critical temperatures is of great importance, to select the right temperature, which will provide large number of nucleation sites for precipitation (dislocation loops, deformation bands, subgrain boundaries, deformation twins) in microstructure during finish rolling. The proper control of critical hot rolling temperatures results in formation of un-recrystallized austenite with high nucleation sites density. In that way, a final fine grained ferritic microstructure forms during continuous cooling from hot deformation temperatures.

Evaluation of microstructural changes during plastic deformation *i.e.* during finish rolling in a hot strip mill is not possible by using traditional quenching of samples and optical microscopy, because it is not possible to quench the sample immediately after the deformation. In order to overcome this problem, it is needed to evaluate changes in microstructures on the basis of behaviour of steel during the deformation. Two opposite processes occur during hot rolling – strain hardening due to deformation and softening due to recovery/recrystallization. Therefore, the stress-strain curve or measured rolling force for each pass can be used to quantify the overall behaviour, based on comparison of yield stress, strain hardening rate and maximal stress. This approach thus quantifies changes in the microstructure based on measured stresses and forces, known as mechanical metallography. It provides accurate and reliable information and enables modelling of microstructural changes in real time on industrial scale.

The aim of this work was to establish the critical temperature for finish hot rolling of microalloyed steels by means of physical simulation.



#### 2. EXPERIMENTAL

The low carbon microalloyed (Nb/Ti) steel tested in this work was industrially melted and casted, and subsequently pre rolled to a sheet, 30 mm thick, in a Hot Strip Mill (HSM) in the company Steelworks Smederevo, Serbia. The chemical composition was provided by the producer and is given in Table 1.

Table 1.	Chemical	composition	of the	tested steel
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Elements	С	Si	Mn	Р	S	Al	Nb	Ti	Ν	0
Content, wt.%	0.06	0.068	0.77	0.015	0.008	0.052	0.039	0.015	0.0053	0.0045

Simulation of hot rolling process during finish rolling was performed by a multi-pass isothermal torsion test, by using a Setaram-Irsid torsion tester (Irsid, France). Main advantage of torsion testing is that it enables attaining high strains without reaching plastic instability [15,19,20]. Furthermore, deformation of several rolling passes can be simulated using only one sample. During torsion, specified deformation values (Table 2) are achieved only on the outer surface of the specimen. On other hand, torsion samples could not be used for metallographic examination, due to occurrence of corrosion and porosity on the outer surface. Isothermal hot rolling simulation was performed to determine critical temperatures. The torsion test specimens were cylinders 6 mm in diameter and 50 mm in gauge length. Specimens were annealed in the testing machine at 1250 °C for 15 min in argon atmosphere, in order to provide homogeneous distribution of alloying elements and to remove the priorly existing texture. Next, the specimens were cooled down to 1000 °C at a rate of approximately 2 °C s<sup>-1</sup>, at which the simulation of roughing was performed, as to provide uniform grain size distribution in all specimens before simulation of rolling at a finishing mill. Simulation of roughing in this test was performed by predeformation (PD1 and PD2 in Fig. 2) in two passes (strain 0.3), with 10 s of the interpass time. These two passes were performed to provide uniform microstructure before the finish hot rolling simulation. Subsequently, the specimens were cooled to the isothermal testing temperature (825, 850, 875 and 950 °C) and held at this temperature for 60 s to provide uniform temperature distribution. Samples were subjected to plastic deformation in five passes. To assess the reproducibility, two tests were run for each set of conditions.



Time

Figure 2. Schematic illustration of the simulation test of thermomechanical processing of microalloyed steel

Details/processing parameters of deformation are shown in Table 2. Strain in each pass is selected to be in accordance with five stand finish rolling schedule in the Hot Strip Mill, Steelworks Smederevo.

Table 2. Schedule	of hot	rolling	simulation
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Pass	-		П		Ш		IV		V
Strain	0.5		0.5		0.35		0.35		0.2
Interpass time, s		4.75		3.25		2.25		1.75	



## 3. RESULTS

Stress-strain curves obtained in torsion tests at specified temperatures are shown in Figure 3.



Figure 3. Experimental stress-strain curves obtained for rolling simulations according to the schedule 4.0 given in Table 2 and the best linear fits in the region of plastic deformation at the testing temperatures



First two passes (0.6 strain in total) at each diagram are related to the roughing rolling stage and will not be discussed in detail. Simulation of finish rolling begins with the third pass, which represents deformation behavior of fully recrystallized samples. In that way, the third pass represents the 1<sup>st</sup> pass of the finish rolling. Stress-strain curves are represented in the graphs in Figure 3 both as experimental points and as the curves obtained by polynomial fitting. The slope of the fitted curve represents a qualitative measure for evaluation of deformation behavior (strengthening or restoration).

As can be seen in Figure 3a, at 950 °C stress-strain curves of each pass obtained during simulation exhibit the same shape, characterized by a high slope of fitted curves in the region of plastic deformation. In comparison, stress-strain curves obtained during simulations at 875 and 850°C (Fig. 3b and Fig. 3c) exhibit lower slopes of fitted curves in the region of plastic deformation while the lowest slope is obtained at 825 °C (Fig. 3d).

In order to determine yield stress ( $\sigma_0$ ), the strain offset method ( $\epsilon \sim 5$  %) was performed, which. provides reliable results according to the literature [13,17]. The yield stress of each finish rolling pass is indicated in the graphs presented in Figure 3.

In order to evaluate and quantify recrystallization during the hot rolling simulation, mechanical metallography was applied, *i.e.* microstructural changes were evaluated on the basis of the change in deformation behavior. Stress-strain curves generated in each pass, during the isothermal torsion test, were compared to each other and analyzed in details. In order to evaluate the progress of softening (recovery and/or recrystallization) during the interpass time, fraction softening (*FS*), was calculated according to the equation [10,13,21]:

$$FS / \% = \frac{\sigma_{\rm m}^{\rm i} - \sigma_{\rm 0}^{\rm i+1}}{\sigma_{\rm m}^{\rm i} - \sigma_{\rm 0}^{\rm 1}} 100 \tag{1}$$

where  $\sigma_m^i$  is the maximum stress in the i<sup>th</sup> pass,  $\sigma_0^1$  is the yield stress in the 1<sup>st</sup> pass,  $\sigma_0^{i+1}$  is the yield stress in the (i+1) pass. All values in equation 1 are determined using the strain offset method ( $\epsilon \sim 5$  %).

According to literature [13,15], *FS* values lower than 20 % indicate recovery as a softening mechanism, *FS* values in range of 20-100 % indicate recrystallization while FS > 100 % indicates grain growth in the material. Furthermore, negative FS values indicate strengthening during interpass time. This strengthening can be attributed to strain induced precipitation [13,15].

An additional attempt to evaluate recrystallization behavior was performed by introducing the area softening parameter (*ASP*) [13]. Area under the curve was calculated by numerical integration. Values obtained in the 1<sup>st</sup> pass were referred as fully recrystallized and were used as a reference for comparison of the areas determined for each pass at equal strains. In order to quantify the SRX behaviour, the area softening parameter (*ASP*) was calculated by using the equation:

$$ASP / \% = \frac{A_{i} - A_{1}}{A_{i}} 100$$
(2)

where  $A_i$  is the area under the stress-strain curve of the i<sup>th</sup> pass and  $A_1$  is the area under the 1<sup>st</sup> curve for the strain level of i<sup>th</sup> pass. *ASP* value lower than 0 % indicates grain growth, while those greater than 0 % indicate strengthening (incomplete static recrystallization). Complete recrystallization is indicated by *ASP* = 0 %.

The determined *FS* and *ASP* values at different temperatures are shown in Figure 4. Estimated standard deviations of calculated FS and ASP values are in the range of 2.4 to 5.7 and 1.8 to 5.1 respectively.





Figure 4. Changes during the interpass intervals described by: (a-d) fractional softening (FS) values between given passes (during the interpass times) and (e-h) area softening parameters (ASP) with passes, at different testing temperatures

### 4. DISCUSION

The aim of performed roughing rolling in this simulation test was to provide uniform grain size distribution as well as fully recrystallized microstructure before the simulation of finish rolling. It was found that after two passes and



subsequent full SRX the influence of the prior austenite grains can be neglected [3,22,23]. It could be observed that the shape of stress-strain curves for the 1<sup>st</sup> pass of finish rolling corresponds to the shape of a stress-strain curve for fully recrystallized conditions [13,16]. Therefore, it could be deduced that before finish rolling, the steel microstructure was recrystallized so that all tested samples had the same starting point in the terms of microstructure.

Shape of the stress-strain curves, strengthening and the restoration process during finish rolling are controlled by interaction of deformation, recrystallization and precipitation processes, *i.e.* by the addition of microalloying elements as well as by hot rolling parameters. Addition of Ti, V, and Nb to low carbon steels, has a great impact on the steel mechanical properties through grain refinement as well as through mechanisms of solid solution strengthening and dispersion strengthening by carbides, nitrides and/or carbonitrides in ferrite [1,8,13-16,24]. Carbides, nitrides and carbonitrides of microalloying elements precipitate at austenite grain boundaries, which cause pinning of austenite grain boundaries at temperatures just over A<sub>3</sub> and retardation/suppression of recrystallization.

Supersaturation ratio, *k*<sub>s</sub>, of niobium carbonitride (Nb(C,N)), defined as the ratio, of actual amount of C, Nb and N in solution to the equilibrium solubility product, at specified temperature was calculated by the equation [24,25]:

$$k_{\rm s} = \frac{c_{\rm Nb} \left( c_{\rm c} + \frac{12}{14} c_{\rm N} \right)}{10^{2.26 - \frac{6770}{7}}} \tag{3}$$

where  $c_{Nb}/$  wt.% is the Nb concentration in steel,  $c_C/$  wt.% is the concentration of C in steel,  $c_N/$  wt.% is the concentration of N in steel, and T/ K is the absolute temperature.

Super saturation ratios for Nb(C,N) on testing temperatures, calculated by eq. (3), are given in Table 3.

Finish rolling temperature, °C	ks	Precipitation
950	4.75	NO
875	10.92	YES
850	14.77	YES
825	20.26	YES

Table 3. Supersaturation ratio of Nb(C,N) at testing temperatures

It is assumed that the critical supersaturation ratio for strain-induced precipitation has to be between 5.5 and 7 [8,26].

Yield stress in passes from 1<sup>st</sup> to 3<sup>rd</sup> at 950°C have similar values, which are forwarded by slight increase in 4<sup>th</sup> and 5<sup>th</sup> pass (Fig. 3a). This behaviour implicates a high share of recrystallization during the interpass times being dominant over the strengthening effect caused by plastic deformation. The obtained phenomena are quantified by calculating fraction softening *FS* values. *FS* values above 80 % obtained at 950 °C indicate almost full static recrystallization (Fig. 4a). Also, the extent of recrystallization is decreasing from 2<sup>nd</sup> to 5<sup>th</sup> pass, which can be attributed to shorter interpass times. The *ASP* values (Fig. 4e) follow the same trend, *i.e.* the increase in *ASP* indicates that SRX was impeded to same extent. Due to the low supersaturation ratio, precipitation is not expected, *i.e.* it can be assumed that the solute drag is responsible for the lower extent of recrystallization. The obtained higher *ASP* values in the last two passes might be explained by accumulation of deformation and a slightly higher level of suppression of recrystallization, which might be dominantly caused by short interpass times.

At 875 °C yield stress of each subsequent pass is higher than the yield stress of the first one (Fig.3b) but difference is not as pronounced as it is at lower temperatures. As can be seen in Figure 3c, each pass during finish rolling simulation at 850 °C has significantly higher yield stress than that obtained in the 1<sup>st</sup> pass as well as in the previous one, which indicates dominant strengthening over softening. Shapes of the obtained curves are in agreement with calculated supersaturation ratio (Table 3). In the test at 875 °C, *FS* values vary around 40 %, indicating low extent of recrystallization (Fig. 4b). Stress-strain curves from the 2<sup>nd</sup> to the final pass exhibit higher yield stress (Fig. 3b) accompanied with lower stress hardening rate which is observed as the lower slope of the fitted curves, shown in Figure 3b. This indicates suppression of recrystallization by stimulation of precipitation of Nb(C,N) [13,14]. Almost constant *FS* at 850 °C from the 1<sup>st</sup> to the last interpass interval might be related to strain-induced precipitation, which strongly affects suppression of



recrystallization [13]. During the 1<sup>st</sup> interpass time some extent of recrystallization takes part, while recovery is dominant during the interpass times between following passes (Fig. 4c). *ASP* values for simulation of hot rolling at 875 °C (Fig. 4f) imply higher strengthening effect from 3<sup>rd</sup> to 5<sup>th</sup> rolling pass compared with *ASP* values obtained at 950 °C. *ASP* results are in agreement with the obtained *FS* values. *ASP* values obtained at 850 °C (Fig. 4g) are higher than values obtained at higher temperatures which indicate suppression of recrystallization and increase in strengthening.

Further lowering of the test temperature to 850 °C (Figs 4c and 4g) and 825 °C (Figs 4d and 4h), induced similar behavior, except for more pronounced suppression of recrystallization. At 825 °C, limited restoration is observed only during the 1<sup>st</sup> interpass interval. It can be assumed that SRX is competing with precipitation, *i.e.* that SRX has a shorter incubation period. On the other hand, once precipitation started, SRX is not the governing phenomenon anymore, *i.e.* precipitation precedes recrystallization, resulting in total suppression of recrystallization. This efficient suppression of recrystallization causes strain accumulation, meaning that due to absence of recrystallization, strength at the beginning of a subsequent pass is equal to already pre-strained condition. In some cases, in the subsequent pass, the critical stress for dynamic recrystallization might be attained [6,28,29]. Results also indicate that dynamic recrystallization does not occur in this work.

Stress-strain curves for the 2<sup>nd</sup> to the 5<sup>th</sup> pass, obtained during simulation of hot rolling at 825 °C (Fig. 3d) show slightly lower values of the yield stress compared to the maximum stress of the previous pass (Fig .3d) and shows higher yield stress compared to yield stress of each previous pass as shown by  $\sigma_0^i$  trendline in Figure 3d. So, it is clear that strengthening is dominant over softening, which is assumed to be recovery [13,15]. Decrease in the yield stress of the 2<sup>nd</sup> pass compared to the maximum strength of the 1<sup>st</sup> pass (*FS* ~20 % Fig. 4d) is assumed to be recovery [13,15]. *FS* values are decreasing from 2<sup>nd</sup> to 5<sup>th</sup> pass (Fig. 4d). *FS* values for 3<sup>rd</sup>, 4<sup>th</sup> and 5<sup>th</sup> are lower than 20 % (Fig. 4d).

The supersaturation ratio of Nb[C,N] (Table 3) at 825 °C (20.26) is higher than 5.5, which indicates intensive straininduced precipitation on austenite grain boundaries [13,15,24,25]. Intensive precipitation causes pinning of grain boundaries and suppression of recrystallization, which agrees with shapes of the stress-strain curves (Fig. 3d) and obtained *FS* values (Fig. 4d) as well as with the *ASP* parameter (Fig. 4h). Suppression of recrystallization causes accumulation of deformation, which results in strengthening of the hot rolled steel.

It can be assumed that due to the high supersaturation ratio, strain accumulation caused strain-induced precipitation during the interpass time, resulting in the higher volume of the precipitated phase and adequate hardening. Based on all results, the critical temperature for the finishing stage of control rolling is estimated to be 825 °C.

#### **5. CONCLUSION**

In this paper, deformation/recrystallization behaviour of microalloyed steel at high temperatures was examined. Steel used in this research was low carbon Nb/Ti microalloyed steel and was tested in order to determine the finish rolling temperature for industrial five stand finish rolling train. Simulation was performed by a multi-pass isothermal torsion test in the temperature range of 825-950°C. Evaluation of microstructural changes during the test was performed by calculation of *FS*. A new method, area softening parameter (*ASP*), for mechanical metallography was also applied to analyse the obtained stress-strain curves.

Individual pass stress-strain curves obtained at 950°C as well as *FS* and *ASP* values indicated full recrystallization during the interpass time. At lower temperatures the extent of recrystallization during the interpass time decreased. At 825°C recrystallization is completely suppressed by strain-induced precipitation, which pins the grain boundaries. Fraction softening and *ASP* values show the same trends for each testing temperature and all the obtained results are in agreement with the shape of stress-strain curves. *ASP* as a criterion provides qualitative results, while accuracy in terms of quantitative analysis should be further developed. Finally, based on all obtained results, the critical temperature for finish rolling was estimated to be 825°C.

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## Sivimulacija završnog valjanja mikrolegiranog čelika u izotermalnim usloma

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(Naučni rad) Izvod

Cilj ovog rada je određivanje temperature završnog valjanja Nb/Ti mikrolegiranog čelika koji sadrži 0,06 % C, 0,77 % Mn, 0,039 % Nb i 0,015 % Ti. Uzorci su ispitivani laboratorijskom simulacijom, na plastomeru - uredjaju za ispitivanje uvijanjem, na temperaturama, između 825 i 950 °C. Režim deformacije u pet provlaka i pauza između provlaka odabrani su u saglasnosti sa parametraima valjanja na petostanskoj završnoj valjačkoj pruzi valjaonice toplovaljanih traka. Udeo obnovljene mikrostrukture (oporavljanja i/ili rekristalizacije) je određivan na osnovu proračuna prekidnog omekšavanja (engl. *fraction softening, FS*) i parametra omekšavanja na osnovu promene površine ispod krive (engl. *area softening parameter, ASP*). Na 950 °C krive deformacionog ojačavanja za svaki provlak, *FS* i *ASP* pokazuju ponašanje koje odgovara potpuno rekristalisanom uzorku. Udeo obnovljene mikrostrukture se smanjuje sa sniženjem temperature do intervala 875-850°C. Pretpostavlja se da zbog velikog presićenja dolazi do pojave taloženja izazvanog deformacijom, što utiče na smanjenje pokretljivosti granica zrna/subzrna što dovodi do potiskivanja rekristalizacije. Na 825°C obnavljanje se odvija isključivo mehanizmom oporavljanja pa se ova temperatura može uzeti za kritičnu temperaturu završnog valjanja. *Ključne reči:* prekidno omekšavanje; mehanička metalografija; kontrolisano valjanje; rekristalizacija, deformacija; kritične temperature

