Influence of rolling speed during hot forming using HDQT-R 30-12 machine on the properties of stainless steel 08Ch18N10T

H. Jirková¹, T. Janda¹, M. Pekovič¹, J. Říha², J. Mach¹ and D. Krsová¹

¹University of West Bohemia, RTI - Regional Technological Institute, Pilsen, Czech Republic, EU

²ŠKODA JS a.s., Orlík 266/15, Pilsen, Czech Republic, EU

E-mail: hstankov@rti.zcu.cz

Abstract. Mechanical properties of austenitic stainless steels can be improved by a suitable combination of forming and heat treatment operations. In addition to rolling reduction and speed, the heat treatment parameters are very important as well. Heat treatment typically combines solution and stabilization annealing. In components of WWER nuclear reactors, most of which are made of these steels, it is necessary to achieve the prescribed hot mechanical properties. Bars of 30 mm diameter made of titanium stabilized 08Ch18N10T steel were rolled in an incremental rolling machine. After induction heating to 950°C, different roll speeds were used, from 200 rpm to 25 rpm. The rolled bars were cooled in water and stabilization annealed at 720°C for 10 hours or at 800°C for 1 hour. Mechanical properties were determined by tensile testing at room temperature and at 350°C. Slower rolling speeds led to lower elongation (40%) than the faster ones (50%). Metallographic evaluation focused on the effect of forming on the grain size.

1 Introduction

Owing to severe thermal and mechanical stresses which act on WWER nuclear reactor components made of titanium-stabilized 08Ch18N10T austenitic stainless steel, this material is required to meet not only strict microcleanliness criteria but also specifications of mechanical properties for the plant project. Although the steel offers very good corrosion resistance and formability, it also exhibits relatively low yield strength [1, 2]. The yield strength required for use in WWER reactor components is at least 177 MPa at 350°C. The basic means for improving mechanical properties are threefold: substitutional or interstitial strengthening of the austenitic matrix, precipitation hardening and work hardening at reduced forming temperatures [3].

It is difficult to use substitutional elements, such as titanium or aluminium, or interstitial elements C and N for strengthening because their amounts are defined by the material specification [2,4]. Titanium is added to prevent Cr₂₃C₆ carbides from forming because the particles deplete the matrix of chromium in their vicinity and make the material more susceptible to intergranular corrosion [2-4]. The strengthening effect is provided by precipitation of carbides and carbonitrides which takes place in the course of heat treatment [3-5]. This heat treatment comprises solution and stabilization annealing. Solution annealing is normally performed in accordance with specifications given in the material data sheet, at 1020-1100°C [5, 6]. Stabilization annealing whose purpose is to improve hot yield strength is carried out at 600-850°C and may take several dozen hours [6, 7].
Work hardening can be induced by cold forming as well as by forming near the lower limit of forming temperatures. Deformation at temperatures no higher than 800°C produces favourable conditions for precipitation-hardening particles to form during subsequent heat treatment. The material develops high dislocation density where the dislocations act as precipitation sites for fine carbonitrides. These precipitates subsequently prevent the movement of dislocations under mechanical load and thus increase the yield strength [3]. By controlling the strain amount and rate along with the forming temperature, one can control dynamic recrystallization of the material which produces dislocation-free equiaxed austenite grains [8]. This steel can be mechanically worked using various processes including forging and rolling. Mechanical properties are governed by the amount and speed of deformation. For these reasons, the present experiment was conducted in HDQT-R 30-12 machine which can be configured to use various roll speeds and heat treatment modules [9].

2 Material and Methods

2.1 Experimental Material
The input stock was 08Ch18N10T steel bars with a diameter of 30 mm and length of 90 mm (table 1). It is a titanium-stabilized austenitic stainless steel. Besides inclusion content, the morphology and distribution of carbides and carbonitrides in this steel are controlled. The steel was delivered in the form of rolled peeled stock.

|       | C     | Cr    | Ni    | Ti    | Mn    | Si    | P     | S     | Cu    | Mo    | V (ppm) | N (ppm) | W     | Co | H (ppm) |
|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|---------|---------|-------|-----|---------|
| C     | 0.05  | 17.75 | 10.05 | 0.43  | 1.8   | 0.52  | 0.024 | 0.014 | 0.1   | 0.08  | 0.11    | 120     | 0.03  | 0.02 | 2.4     |

2.2 Incremental Rolling
Bars of 900 mm were processed in incremental rolling machine HDQT–R30–12 (High Deformation Quenching and Tempering) (figure 1). This rolling mill comprises three conical rolls for helical rolling whose axes are inclined relative to each other at 120°. The rotational movement of the rollers causes the formed bar to be helically drawn into the cylinders. Incremental reduction in the bar diameter is thus applied across its entire length [10]. Prior to forming, the bar stock is heated in inductor coils. The heating temperature is controlled by the inductor power settings. Prior to entry into and exit from the rollers, the temperature of the bars is measured with a pyrometer.

Figure 1. Helical rolling mill (HDQT - R 30-12) at Regional Technological Institute [9]

The process routes began with heating the bars to 970°C. Heating was followed by helical rolling. The first pass reduced the stock diameter from 30 mm to 25 mm and the second one produced a diameter of 21 mm. The roll speed was varied from 200 to 25 rpm. The impact of the amount of reduction on grain size was explored by incorporating a route in which the diameter was reduced
from 30 mm to 21 mm in a single pass at a roll speed of 100 rpm. The rolled bars were cooled in a water tank at room temperature placed at the end of the roller track. By this operation, the process equivalent to solution annealing was effectively completed.

In order to improved mechanical properties, the products were then stabilization annealed using three different routes. The first one was a standard route, at 720°C for 10 hours. The second one was a combined process: 680°C for 5 hours + 720°C for 10 hours. The last route with an increased temperature and much shorter time, 800°C and 1 hour, respectively, was tested in order to explore the potential for time and energy savings.

2.3. Methods of Evaluation
Metallographic characterization on longitudinal sections through the bars was performed under an Olympus optical microscope. The evaluation focused on the condition of as-received bars as well as the results of the rolling and heat treatment routes. The grain size near the surface and along the axis of the bar stock was determined using the intercept method according to ASTM A112 [11]. Vickers hardness was measured in the same locations (HV10) according to EN ISO 6507-1:2018 [12]. Tensile tests on test bars 10 mm in diameter and 50 mm in length were carried out at ambient temperature according to EN ISO 6892-1 [13] and at 350°C according to EN ISO 6892-2 [14].

3 Results and Discussion
The initial microstructure comprised austenite grains which were distorted by rolling (figure 2a). The grain size was 9.7 µm below the surface of the bar and 10.7 µm along the bar's axis. Slip bands and titanium carbides and carbonitrides were present. The as-received bars had a surface hardness of 247 HV10 and a hardness of 246 HV10 in the centre.

3.1. Effects of roll speed
Rolling of stock heated to 970°C at various speeds led to a microstructure of fine austenite and typical features including twins, slip bands and titanium precipitates. Rolling at 200 and 100 rpm (R200, R100) led to dynamic recrystallization which produced equiaxed austenite grains (figure 2b). The grain size was between 9.7 and 12 µm (table 2). The expectation that recrystallization led to softening was confirmed by hardness testing. Hardness was lower than in the as-received condition: 145 HV10 near the surface of the bar and 142 HV10 along its axis (table 2). Values were similar for the 100 rpm speed and for the single-pass reduction from 30 to 21 mm (R100-1).

| Roll speed (rpm) | Number of passes | Temperature at exit from the rolls (°C) | Grain size (µm) | HV10 (-) |
|------------------|------------------|----------------------------------------|----------------|----------|
|                  |                  | Surface  | Axis   |                    | Surface  | Axis   |
| IS               | -                | -        | 9.7  | 10.7  | 247 | 246 |
| R200             | 200              | 2        | 1020 | 9.7  | 10.3 | 145 | 142 |
| R100             | 100              | 2        | 971  | 9.5  | 10.8 | 144 | 141 |
| R75              | 75               | 2        | 963  | 9.3  | 10.0 | 165 | 163 |
| R50              | 50               | 2        | 925  | 9.7  | 10.3 | 184 | 184 |
| R25              | 25               | 2        | 846  | 11.5 | 11.5 | 229 | 200 |
| R100-1           | 100              | 1        | 1017 | 9.6  | 10.8 | 145 | 143 |

At roll speed of 75 rpm (R75), only partial recrystallization took place, as evidenced by a relatively high hardness: 165 HV10. Slower rolling at 50 rpm (R50) and 25 rpm (R25) failed to support dynamic recrystallization and led to a microstructure of elongated austenite grains (figure 2c). This was reflected in the further increase in hardness to 229 HV10 near the surface of the bar. Slower roll speeds resulted in a slightly larger austenite grain: 11.5 µm (table 2). The higher hardness obtained at the speed of 25 rpm was the result of low finish-rolling temperature due to the long contact between...
the bar and the water-cooled rolls. At the exit from the rolling mill, the temperature was 846°C. The highest temperature at the exit from the rolls was 1020°C, which was found at roll speed of 200 rpm. Rolling speed had an impact on mechanical properties as well. The ultimate tensile strength at room temperature was 580 MPa for roll speeds of 200 and 100 rpm. Yield strength exceeded 220 MPa and elongation was higher than 50% (figure 3a). Lower elongations, by approximately 10%, were found after routes with slower rolling. The corresponding yield strengths were higher, in excess of 300 MPa, as were the ultimate strengths; more than 610 MPa.

Tensile test at 350°C on materials rolled at higher speeds 100 through 200 rpm revealed ultimate strengths of approx. 460 MPa, yield strength of more than 160 MPa and elongations higher than 36% (figure 3b). From the route with the roll speed of 75 rpm onwards, the elongation levels decreased to 31 and 30% but yield strength increased to 296 MPa at the roll speed of 25 rpm (figure 3b).

3.1. Stabilization annealing

With a view to improving mechanical properties, namely hot yield strength, the rolling operation was followed by stabilization annealing at 720°C for 10 hours, two-step stabilization annealing at 680°C/5 hours + 720°C/10 hours and an energy-saving annealing cycle at 800°C for a mere hour.

After stabilization annealing, the microstructure of the bars which had been rolled at 200 and 100 rpm speeds consisted primarily of recrystallized grains with precipitates along boundaries and within austenite grains (figure 4a). No microstructural differences were found among the products of the stabilization annealing cycles, including the grain size which was approximately 9 µm in all cases. After rolling at 75 rpm, recrystallized microstructures were only obtained by combined annealing. After annealing at 720°C/10 hours, the microstructure was not fully recrystallized and contained some regions with very fine grain (figure 4b). A short hold at the higher temperature (800°C) failed
to eliminate distortion introduced by rolling. After rolling at the slowest speed, 25 rpm, the distorted grain structure remained unchanged even after stabilization annealing (figure 4c).

![Figure 4. Microstructure near the bar’s surface after stabilization annealing at 720°C/10 hours: a) 200 rpm, b) 75 rpm, c) 25 rpm](image)

Figure 5. Results of tensile testing at 350°C for stabilization-annealed material at different temperatures

The effects of various stabilization annealing procedures were also reflected in mechanical properties measured by tensile test at 350°C. After the standard route with a heating temperature of 720°C and a holding time of 10 hours, the yield strength was above the required value of 177 MPa in all the cases (figure 5). The ultimate strength was above 440 MPa, with elongation at 35%. In bars rolled at 50 or 25 rpm, yield strength was higher, in the range of 298 to 317 MPa. Elongation was, however, lower than before, by approximately 30%.

Rolling at the highest speeds and combined stabilization annealing led to yield strengths of less than the specified 177 MPa. As in the previous case, yield strength increased with decreasing roll speed. At the lowest speed, 25 rpm, it was 349 MPa. This route, however, also led to the lowest elongation level of 24%. The energy and time-saving route with a heating temperature of 800°C and 1-hour holding time produced the lowest yield strength, regardless of the roll speed. The specified yield strength was only achieved at and beyond the speed of 75 rpm. These results show that the conventional stabilization annealing route, 720°C/10 hours, is a reliable way to achieve the prescribed mechanical properties regardless of the speed of the prior rolling process.

2 Conclusion
In this experimental study, the effects of roll speed were explored on 08Ch18N10T steel bars processed in an incremental rolling mill. The impact of stabilization annealing on mechanical properties was examined as well.
It was found that at roll speeds of 75 rpm or less, yield strength increased significantly. At 25 rpm, the increase was up to 130 MPa when compared to the highest roll speeds. These results are in agreement with the increase in hardness from 145 HV10 to 229 HV10. The stock passed through the rolls more slowly and was therefore cooled down by the contact with water-cooled rolls. This was reflected not only in mechanical properties but also in the structure of the product which consisted of elongated austenite grains. In all the products, stabilization annealing at 720°C for 10 hours produced hot yield strengths of more than the specified limit of 177 MPa. The values ranged from 181 MPa for roll speed 200 rpm to 317 MPa for roll speed 25 rpm. Combined annealing and the energy-saving stabilization annealing only led to acceptable yield strengths with roll speeds of 75 rpm and less.

Acknowledgements
This research was funded by the Technology Agency of the Czech Republic under the project TJ02000274 „Determination of the principles and processes taking place during the stabilization annealing of austenitic stainless steels used in nuclear power “.

References
[1] Pardo A, Merino M C, Coy A E, Viejo F, Arrabal R and Matykina E 2008 Effect of Mo and Mn additions on the corrosion behaviour of AISI 304 and 316 stainless steels in H2SO4 Corrosion Science 50(3) 780–794
[2] Rezaei H A, Ghazani M S and Eghbali B 2018 Effect of post deformation annealing on the microstructure and mechanical properties of cold rolled AISI 321 austenitic stainless steel Materials Science and Engineering A 736 364–374
[3] Petman I. Některé problémy výroby vysokolegovaných ocelí pro komponenty jaderných elektráren typu VVER 440. 1980 Conference proceedings: Materiálové problémy lehkovodních reaktorů, Praha: Dům techniky ČSVTS pp. 22-27
[4] Wang J, Su H, Chen K, Du D, Zhang L and Shen Z 2019 Effect of δ-ferrite on the stress corrosion cracking behavior of 321 stainless steel Corrosion Science 158 108079
[5] Moura V, Kina A Y, Taveres S S M, Lima L D and Mainier F B 2008 Influence of stabilization heat treatments on microstructure, hardness and intergranular corrosion resistance of the AISI 321 stainless steel Journal of Materials Science 43(2) 536–540
[6] Kaneko K et al. 2011 Formation of M23C6-type precipitates and chromium-depleted zones in austenite stainless steel Scripta Materialia 65(6) 509–512
[7] Podaný P, Martinek P, Nacházel J and Balcar M Heat Treatment of Reactor Vessel Steel 08Ch18N10T. 2012 MS&T 2012: Materials Science and Technology Conference and Exhibition 2012. NY: Materials Science and Technology (MS&T) pp. 1036–43
[8] Peković M, Vrtáček J, Janda T and Volkmanová J Induction hardening of steels with use of the device for incremental forming of round bars HDQT-R 30-12 2020 IOP Conf. Ser.: Mater. Sci. Eng. 723 012025
[9] Ghazani M S and Eghbali B Characterization of the hot deformation microstructure of AISI 321 austenitic stainless steel Materials Science and Engineering: A 730 380–390
[10] Peković M. et al. 2018 Thermomechanické zpracování ocelí s použitím zařízení pro inkrementální tváření tyčí HDQT-R 30-12 (Thermomechanical treatment of steels with using the device of incremental bars forming HDQT-R 30-12) Kovárenství 65 26-29
[11] ASTM E112 - Standard Test Methods for Determining Average Grain Size, 2013
[12] EN ISO 6507-1 Metallic materials - Vickers hardness test – Part 1: Test method, 2018
[13] EN ISO 6892-1 Metallic materials – Tensile testing – Part 1: Method of test at room temperature, 2020
[14] EN ISO 6892-1 Metallic materials – Tensile testing – Part 1: Method of test at elevated temperature, 2018