EFFECT OF HOLDING TIME AT STABILIZATION ANNEALING ON PROPERTIES OF 08CH18N10T AUSTENITIC STAINLESS STEEL

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Abstract

Titanium-stabilized austenitic stainless steel 08Ch18N10T is often used in nuclear power facilities due to its favourable properties. Many parts of the primary circuit of VVER 440 and VVER 1000-type nuclear power plants are manufactured of this steel. This means that the material must possess very good corrosion resistance and thermal stability of structure. Yield strength of at least 177 MPa at 350 °C is required. Heat treatment of this steel comprises several important stages which affect the resulting mechanical properties. In particular, the solution and stabilization annealing after forging or rolling are time and energy consuming. Therefore, various ways to optimize these processes are being sought.

A combination of solution annealing at 1020 °C for 15 and 30 minutes and stabilization annealing at 720 °C with dwell of 2.5 hours to 15 hours was tested on 08Ch18N10 steel samples. The aim was to establish the effects of stabilization annealing and solution annealing times on mechanical properties. Longer solution annealing has a pronounced effect on the reduction of scatter in mechanical properties. All annealing routes led to yield strengths at 350 °C higher than 200 MPa and tensile strengths of more than 380 MPa.

Keywords: 08Ch08N10T, solution annealing, stabilization annealing

1. INTRODUCTION

The purpose of the heat treatment of austenitic stainless steels is to obtain desired mechanical and corrosion properties and, where relevant, reduce internal stresses. Thanks to their creep resistance and resistance to intergranular corrosion, they are often used in applications involving elevated temperatures. In WWER nuclear plants, one prominent representative of this class of steels is the GOST 5632 grade. Its properties comply with the former Soviet design code for nuclear facilities PNAE-G. Its structure is stabilized with titanium. Its alternative designation is 08Ch18N10T. The properties of this steel strongly depend on titanium carbide and carbonitride precipitates which form during stabilization annealing [1]. The purpose of stabilization with titanium is to prevent precipitation of $Cr_{23}C_6$ carbides, which deplete the matrix of chromium [2,3]. Furthermore, these carbides precipitating on grain boundaries reduce the material resistance to intergranular corrosion. In steels of this type, this difficulty is eliminated by stabilization annealing at 845–950 °C [4,5].

The full heat treatment sequence for this steel consists of solution and stabilization annealing. Solution annealing is performed in accordance with specifications in the temperature ranges from 1020 to1100 °C [1,2] or from 1100 to 1150 °C for 1 to 5 hours [6-8]. A temperature of 1200 °C is used only exceptionally [8]. The subsequent stabilization annealing leads to increased mechanical properties, particularly hot yield strength. It is performed at the temperatures from 600 to 850 °C with dwelling times up to tens of hours [1,6]. The task of treating the 08Ch18N10T steel for nuclear power applications correctly is rather complex. The previous treatment history needs to be known as well because it governs the outcomes of both solution and stabilization annealing.

The difficulty with heat treating this steel grade is the long times and the amount of energy required. This is why alternative optimum combinations are sought, involving solution annealing and stabilization annealing with shorter times, particularly in the latter, while achieving compliance with prescribed mechanical properties. In this paper, description is given of the effects of the stabilization annealing time on hot yield strength in 08Ch18N10T steel. Stabilization annealing followed after solution annealing with different annealing times.

2. MATERIALS AND METHODS

2.1. Experimental material

The experiments outlined below were performed on 08CH18N10T steel (**Table 1)**. In addition to inclusion content, the microstructure of this steel is monitored for the content, morphology and distribution of titanium carbonitrides. The relative levels of carbon and titanium are important as well. There is a specified value of hot yield strength for use in WWER reactor facilities: at 350 °C it should be no less than 177 MPa. An equivalent grade is AISI 321 according to ASTM/ASME (the designation in EN standards is X6CrNiTi18-10 (1.4541)). However, the minimum prescribed hot yield strength for this equivalent grade is less than the requirement for WWER facilities.

Table 1 Chemical composition of 08Ch18N10T steel in the present experiment (wt%)

C	Cr	Ni	Ti	Mn	Si	P	S	Cu	Mo	V	N (ppm)	W	Co	H (ppm)
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

The steel was supplied as heat-treated bars 90 mm in diameter. The heat treatment route comprised solution annealing at 1020 °C for 110 minutes and stabilization annealing at 720 °C for 600 minutes. The bars were sectioned by waterjet cutting into samples 45 × 20 × 110 mm in size. These were then experimentally heat-treated using various parameters.

2.2. Heat treatment

The treatment was performed in an air furnace. The solution annealing temperature was 1020 °C. As the samples were much smaller than the bars, shorter times at temperature were used: 15 minutes and 30 minutes after the temperature in the entire sample became homogeneous. The temperature was measured with a K-type thermocouple placed in a hole drilled in the sample to a depth of 10 mm. The annealing temperature could thus be monitored, as well as the time required for bringing the sample to temperature. After solution annealing, the samples were quenched in water. Stabilization annealing at 720 °C involved five different times from 2.5 hours to 15 hours. The samples then cooled in air. As with solution annealing, the ramp time for reaching the annealing temperature was measured using thermocouple.

2.3. Methods of evaluation

The microstructures of the as-received material (initial state) and the treated samples were documented using an Olympus optical microscope (OM). Detailed examination, including a chemical analysis using EDS, was performed in Zeiss EVO MA and Tescan Easy Probe scanning electron microscopes (SEM). Mechanical properties were determined by tensile testing at room temperature (RT) according to EN ISO 6892-1 and at 350 °C in accordance with EN ISO 6892-2. The diameter and gauge length of the test pieces were d = 10 mm and l_0 = 50 mm, respectively. In each case, two samples were used for tensile testing, one located near the surface of the original bar (referred to as “edge”) and one from the central region (“centre”) because austenite grain size was expected to vary across the bar’s cross section. From each sample, two tensile test pieces were made and specimens for microstructure characterization were taken. Hence, the impact of the treatment as

well that of the location within the original bar to be assessed. In addition, hardness was measured and reported using HV10 hardness number.

3. RESULTS AND DISCUSSION

3.1. Microstructure analysis

The microstructure of as-received bars of 90 mm diameter was examined on longitudinal and transverse cross sections. It contained equiaxed austenite grains with distinct slip bands, twins and intermetallic phases, including titanium nitrides, carbides and carbonitrides, and a small amount of delta ferrite (**Figure 1**). The macrostructure was non-uniform on the cross section, consisting of coarser and finer austenite regions, with the grain size increasing slightly toward the centre of the bar. This was confirmed by grain size measurement using the intercept method according to ASTM A112. In the "edge" sample, the grain size was 44 and 54 µm on the longitudinal and transverse cross sections, respectively. In the "centre" sample, it was 63 and 60 µm on the longitudinal and transverse cross sections, respectively (**Figure 2**). Solution annealing for 15 and 30 minutes caused no appreciable changes in the microstructure (**Figure 3**). The austenite grain size remained substantially unchanged as well (**Figure 2**).

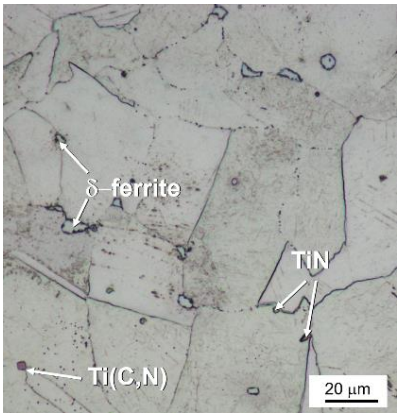

Figure 1 Microstructure of as-received bars, "centre", optical micrograph

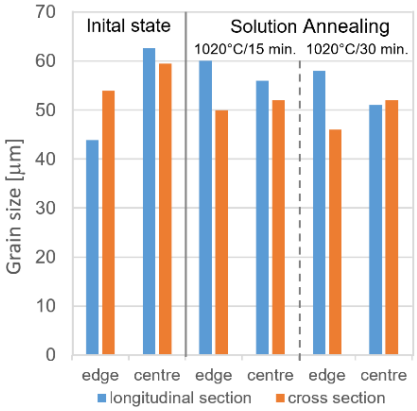

Figure 2 Grain size of as-received material (initial state) and the solution-annealed samples

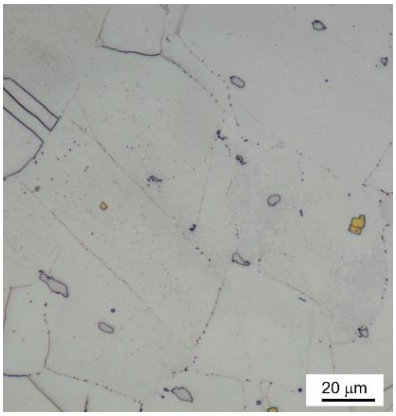

Figure 3 Optical micrograph of solution annealed material – 1020 °C/30 minutes

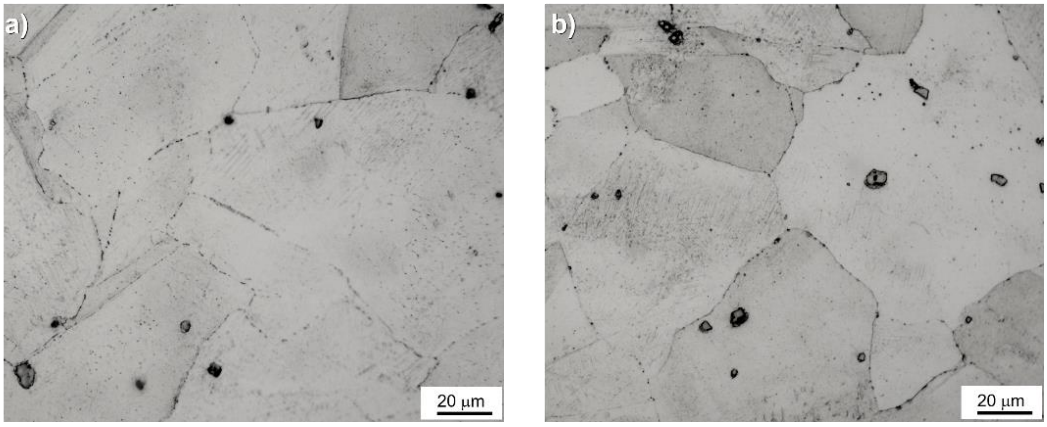

Figure 4 Micrographs of stabilization-annealed material, 720 °C (after solution annealing 1020 °C/30 minutes): a) time at temperature: 2.5 hours, b) 15 hours

The variations with the location within the bar could be attributed to the non-uniformity of the initial condition of the material. Yet, stabilization annealing led to minute structural changes. Precipitates became coarser and a dispersion of fine particles emerged within austenite grains (**Figure 4**).

3.2. Mechanical testing

Mechanical tests were performed at room temperature and at 350 °C. The requirement for parts of WWER-type nuclear facility made of this steel is hot yield strength (at 350 °C) of no less than 177 MPa. Testing was performed on "edge" samples located near the surface of the original bar and "centre" samples close to the axis of the bar.

The ultimate strength at room temperature in "edge" samples was 573 MPa. This was higher than in the "centre": 560 MPa. Hot yield strength in "edge" and "centre" samples was 337 MPa and 335 MPa, respectively. These values correlate with grain size which was finer near the bar's surface than in the centre (**Figure 5**). Given the size and non-uniformity of the structure across the cross section, the differences of 13 MPa in ultimate strength and 2 MPa in yield strength are negligible and have no practical impact. Testing at 350 °C led to an ultimate strength of 410 MPa, yield strength of 276 MPa and elongation of 26% (**Figure 5**). After solution annealing, the ultimate strength and yield strength were lower. The ultimate strength at room temperature was 30 MPa lower after annealing for 15 minutes. Annealing for 15 minutes caused yield strength to decrease from 336 MPa to 270 MPa. The 30-minute annealing led to a value of 293 MPa. Elongation rose by approximately 5 percentage points. The likely causes of this softening include dissolution of fine intermetallic particles and reduction in dislocation density. Similar trends were found by tests at 350 °C. After solution annealing, the yield strength decreased to 207 and 230 MPa. In both cases, the criterion of the minimum value of 177 MPa was met.

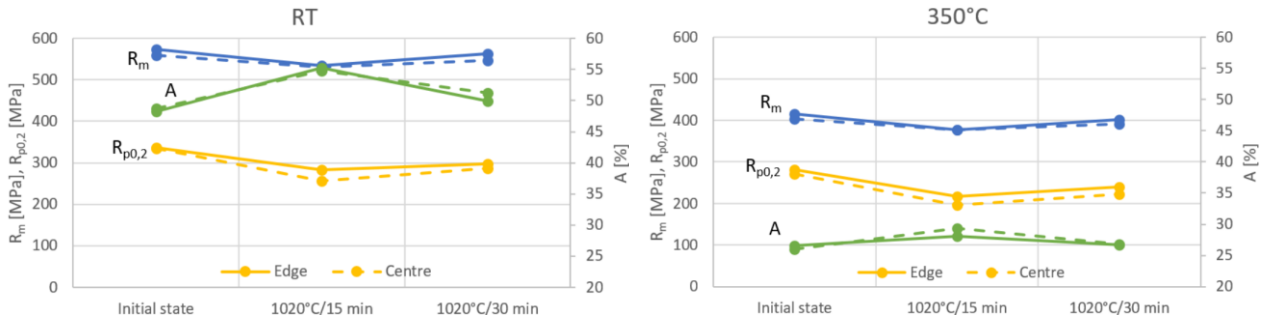

Figure 5 Mechanical properties of as-received material (initial state) and solution-annealed samples tested at RT and 350 °C

Testing at room temperature after stabilization annealing at 720 °C revealed that longer times at temperature lead to higher ultimate strength and yield strength. The impact of the preceding step, i.e. solution annealing at 1020 °C for 15 and 30 minutes, was assessed as well. After 15-minute solution annealing, longer times of stabilization annealing at 720 °C led to higher ultimate strengths. Expressed numerically, 540 MPa was obtained after 2.5-hour annealing, whereas 560 MPa was found after 15 hours (**Figure 6**). Yield strength showed the same trend. This means that longer times at 720 °C resulted in increased yield strength: from 295 MPa to 330 MPa. By contrast, elongation decreased slightly with increasing time at temperature: from approximately 54 % to 51 %.

Solution annealing with longer time at temperature, 30 minutes, combined with the subsequent stabilization annealing led to ultimate strengths from 541 to 545 MPa. Yield strengths were between 294 and 306 MPa (**Figure 6**). Elongation values were around 53 %, regardless of the length of stabilization annealing. The difference between the "edge" and "centre" samples was minute: under 10 MPa.

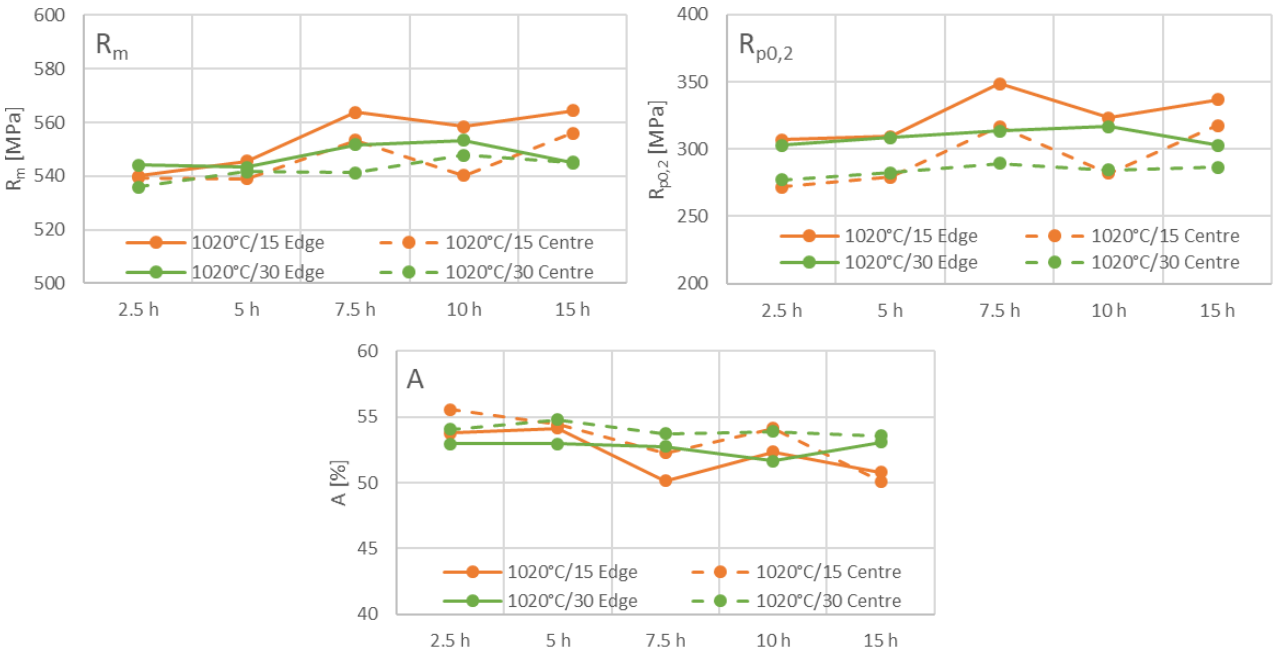

Figure 6 Results of tensile testing at room temperature for stabilization-annealed material and different times at temperature

Tensile testing at 350 °C revealed no substantial impact of the time at stabilization annealing temperature (**Figure 7**). Slight increases were found after combinations with 15-minute solution annealing. With longer annealing times, the ultimate strength rose from 390 MPa to 404 MPa and the yield strength from 238 MPa to 260 MPa. When the preceding solution annealing time was 30 minutes, there was no change in these values with stabilization annealing time. In all the cases, the yield strength was higher than the specified minimum of 177 MPa. Longer solution annealing times clearly led to more consistent properties after stabilization annealing. After those routes which involved the longer solution annealing time, there were no dramatic variations between specimens.

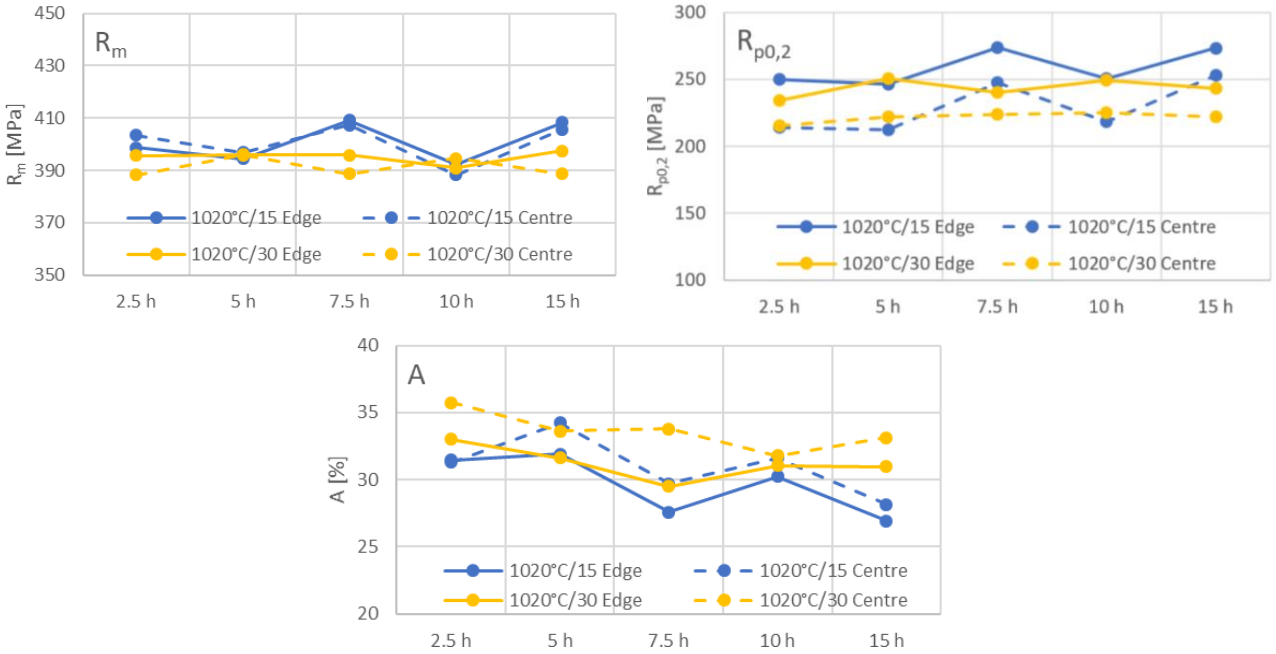

Figure 7 Results of tensile testing at 350 °C for stabilization-annealed material and different times

4. CONCLUSION

Austenitic stainless steel grade 08CH18N10T stabilized with titanium was used to investigate the effect of stabilization annealing times at 720 °C on mechanical properties. Stabilization annealing was preceded by solution annealing at 1020 °C for 15 minutes or 30 minutes and by water quenching. The dwelling times at the temperature of stabilization annealing were from 2.5 to 15 hours.

The resulting microstructure consisted of equiaxed austenite grains with numerous annealing twins, delta ferrite grains and intermediate phase particles (Ti(C,N), TiN). These phases were found within austenite grains as well as at their boundaries. The ultimate strength and yield strength at room temperature increased slightly with time at the stabilization annealing temperature of 720 °C. Notably higher values were obtained after routes with 15-minute solution annealing. The ultimate strength was 540 - 560 MPa at room temperature and 391 – 404 MPa at 350 °C. The yield strength ranged from 295 MPa to 330 MPa at room temperature and from 238 MPa to 267 MPa at 350 °C. Elongation $A_{5.65}$ was 51 % - 54 % at room temperature and 27 % to 34 % at 350 °C. Results without dramatic variation between specimens were obtained with the longer solution annealing time. Longer times at 1020 °C produce more uniform structure and consistent results.

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