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Research Article

Determination of Thermomechanical Processing Parameters of Cold Rolled AISI 441 Stainless Steel

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Abstract

AISI 441 stainless quality steel is a material with high thermal conductivity and suitable for hot working conditions. Due to these properties, it is widely used in the automotive industry, exhaust systems and heat exchangers. AISI 441 is an alloy specially produced for these applications. Titanium and niobium additions prevent grain growth and segregation at high temperatures while increasing pitting corrosion resistance. The relatively high niobium content provides oxidation resistance at high temperatures. Due to its high chromium, titanium and niobium content, it can show faster secondary hardening than other ferritic grades. Therefore, thermomechanical process parameters require more precise adjustment. In this study, the parameters of cold rolling, continuous heat treatment and pickling processes of AISI 441 were investigated. The mechanical properties and corrosion resistance of the produced alloy were investigated.

Introduction

Stainless steel is an engineering material of increasing economic importance in the world. Its chemical stability, corrosion resistance and ability to be shaped have made austenitic stainless steels stand out since its discovery. Today, increasing raw material prices and fluctuating nickel prices, which is one of the most important alloying elements of austenitic stainless steel, have led the market and R&D studies to ferritic stainless steels [1]. AISI 441 cold rolled stainless steel offers many advantages thanks to its production in accordance with EN 10088-2 standard. This standard specifies that the steel complies with high quality and reliability standards. AISI 441 grade stainless steel has an increasingly wide range of uses. It is especially preferred in various sectors such as exhaust construction in the automotive sector, kitchenware, industrial coatings, temperature-controlled environments and even building materials. The mechanical strength, corrosion resistance and high temperature performance of AISI 441 make this steel ideal for use in particularly harsh operating conditions [2].

Below are the chemical composition limits of AISI 441 alloy according to EN 10088-2 standard [3].

Table 1: Chemical properties of AISI 441 alloy

CHEMICAL COMPOSITION (BY WEIGHT A%)								
C	SI	P	S	CR	MO	NI	N	NB
0.030	1.00	0.040	0,015	17,5-18,5	-	-	-	[3xC+0,30]-1,00

When the chemical composition is examined, the alloy containing titanium and niobium, which are micro-alloy elements, puts AISI 441 in a separate place in the ferritic stainless-steel group. By using dual stabilisers, mechanical strength, chemical strength, weldability and machinability were tried to be increased [4, 5]. The titanium in its content increases corrosion resistance by increasing its passivisation capability. It also increases its stabilisation by increasing its oxidation resistance at high temperature. It reinforces its high temperature usage properties by increasing its thermal expansion resistance in accordance with the place of use [6]. The addition of niobium improved the relatively poor weldability of ferritic stainless steels by increasing weldability. In addition, by preventing carbide precipitation, it prevents sensitisation of the steel at high temperatures. With this feature, it increases the high temperature strength and prevents defects such as hot cracks and carbide spalling in the welding process [7].

Table 2: Mechanical properties of AISI 441 alloy

Yield Strength Rp0.2 min.	Tensile Strength	Elongation Amount min.
230 MPa	430-630 MPa	18%

The above table shows the mechanical property limits of cold rolled, solution annealed, mechanically tempered AISI 441 alloy according to TS EN 10088-2 standard. 2B surface quality stainless steel plate production flow is given below

Figure 1: Stainless steel plate production flow

he cold rolling process is the process of reducing the hot rolled semi-finished product to thinner thicknesses by cold rolling. Cold rolling offers many advantages such as material production in thinner thicknesses, higher and aesthetic surface quality. Cold rolling of high strength materials is usually done with 20 roll rolling machines. Figure 1 shows the roll arrangement of the 20-roll rolling machine [8].

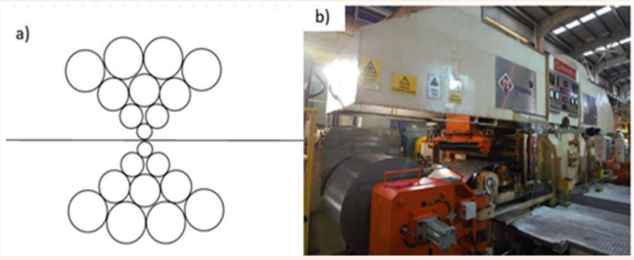

Figure 2: 20 roll cold rolling machine

During cold deformation of materials, changes occur in their internal structure and phases. Grains break and elongate. In this way, hardness, yield strength and tensile strength increase while corrosion resistance and plastic deformability decrease. The material produced here is labelled as 2H according to TS EN 10088-2 as surface quality [9]. Grains are recovered in the dissolution heat treatment [8]. The material is taken into the desired phase solution. In this way, yield strength, tensile strength, hardness and plastic deformation properties are brought to the limits required by the industry. During this heat treatment, high temperature oxides are formed on the surface. The removal of these oxides with acid mixtures and the surface passivation of stainless steel is called pickling process [10, 11]. These processes are carried out in continuous heat treatment and pickling lines. As a result of these processes, the material is produced with 2D surface quality. AISI 441 solution heat treatment has been investigated in many studies due to its Ti and Nb content. The solution heat treatment ranges between 950 °C and 1050 °C depending on the other processes of continuous annealing and pickling and the optimised value. Pickling is the process of removing the high temperature oxides formed during annealing on stainless steel and forming a passive oxide film (Cr2O3) on the surface thanks to the chromium element in the structure. The increase in annealing time in continuous heat treatment lines also increases the pickling time depending on the line speed. Therefore, the first and biggest constraint to efficient production is pickling speed. Pickling of ferritic stainless steels is more difficult due to the instability of the main phases. Therefore, it involves many combined processes [12]. ASM Metal Handbook - Surface engineering book states that the prerequisite for obtaining a clean surface on ferritic grade stainless steels is preferably melt salt bath treatment and bipolar electrolysis [13]. Furnace parameters are very important in stainless steel continuous annealing and pickling, especially in ferritic grades. In the mechanical tempering process, the material is rolled in the elastic zone with smooth, thick rollers without thinning. In this process, the surface roughness of the material decreases and the surface becomes brighter [14]. In this study, the production stages of AISI 441 grade stainless steel flat products were examined and the mechanical and intergranular corrosion resistance of the stainless steel produced were examined in accordance with the A262 standard.

Experimental Studies

The experimental studies are analysed below.

Material Selection

The chemical properties of the produced AISI 441 stainless steel are given in the table below. For the production of 2B surface, 1D surface quality (hot rolled and acid cleaned) 3 mm. thick 21 tonne rolls were used.

Table 3: Chemical properties of AISI 441 alloys

Alloy	Coil Number	Width (mm)			CHEMICAL COMPOSITION(A%)							
			C	Si	Mn	P	S	Cr	Ni	Ti	Nb	N
441	F202436	1290	0.01	0.3	0.3	0.03	0.0005	17.09	0.1	0.2	0.4	0.01
441	F202437	1286	0.01	0.3	0.3	0.03	0.0005	17.09	0.1	0.2	0.4	0.01
441	F202439	1290	0.01	0.3	0.3	0.03	0.0005	17.08	0.1	0.2	0.4	0.01
441	F202440	1288	0.01	0.3	0.3	0.03	0.0005	17.08	0.1	0.2	0.4	0.01
441	F202442	1287	0.01	0.3	0.3	0.03	0.0005	17.08	0.1	0.2	0.4	0.01

Cold rolling

With the cold rolling machine in Trinox Metal plant, 22 tonnes of AISI 441 grade stainless steel material with a thickness of 3 mm and a width of 1280 mm was cold rolled to 0.39 mm (81.9%). This machine is a Tenova I2S, Sendzimir model and has a rolling system with 20 rollers, the machine used is shown below. As a rolling method, it is also known as extrusive rolling mill because of the importance of tension in this type of machine. The material was deformed 81,9% without intermediate annealing.

Table 4: Cold rolling process parameters

Alloy (AISI)	Raw Material Thickness (mm)	Finish Thickness (mm)	Width (mm)	Pass Number	Total Deformation (%)
441	3	0,39	1280	11	81,9

Continuous heat treatment and pickling

After cold deformation, the mechanical properties of the materials were tried to be brought to the desired level by solution annealing.

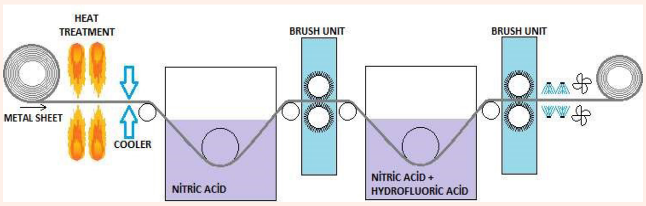

Figure 3: Schematic representation of continuous heat treatment line

The schematic representation of the continuous heat treatment line is given in Figure 3. Heat treatment and surface treatment parameters of the obtained samples are given in the table below.

Table 5: Heat treatment and pickling parameters

Alloy (AISI)	Thickness (mm)	Furnace Temperature (°C)	Heat Treatment Time (s)	Descaling Time (s)	Electro Pickling Time (s)	Final Pickling Time (s)
441	0.39	950	100	33	66	66

The schematic representation of the continuous heat treatment line is given in Figure 3. Heat treatment and surface treatment parameters of the obtained samples are given in the table below. Solution ratios are given by weight.

Table 6: Surface treatment parameters

Alloy (AISI)	Thickness (mm)	Descaling	Electro Pickling	Final Pickling	Electrolysis (A/h)	Solution Temperature (°C)
441	0.39	15% H_2SO_4 + 2% HCl	15% HNO_3 + Electrolysis	15% HNO_3 + 2% HF	3000+2500	55-60

Elektropickling

The electropickling process is carried out by immersing the oxide layer on the surface of the material into acid or neutral salt solution by electrochemical reactions with anode and cathode electrodes. It is the process of cleaning the surface by dissolving the oxide layer on the material surface with the current supplied to the electrodes. The schematic representation of the electropickling unit is shown in Figure 4. The related process is called bipolar electrolysis or inter-electrolysis process [15].

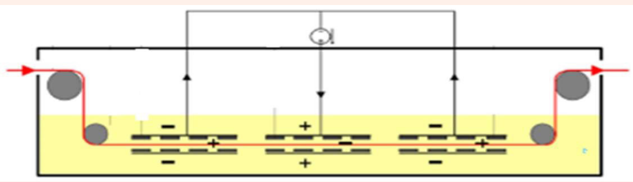

Figure 4: Schematic representation of the electropickling unit

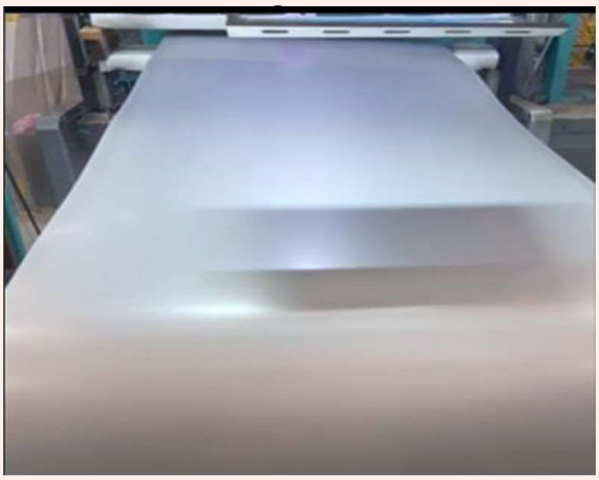

Figure 5: Material visualisation after continuous heat treatment and pickling

Skinpass Tempering Mill

After heat treatment and pickling, the material was brought from 2D surface quality to 2D surface quality and the planarity of the material was ensured after tempering. Tempering parameters of the obtained samples are given in the table.

Table 7: Tempering parameters

Alloy (AISI)	Thickness (mm.)	Width (mm)	Pass Number	1st Pass (Tonne)	2nd Pass (Tonne)	3rd Pass Baskı (Tonne)	Tension (Tonne)
441	0.39	1280	3	10.4	4.2	2.2	10,2

Cup Test

In order to determine the deep drawing ability of the material after tempering during forming, cup test was performed according to ASTM E643 standard. Especially in ferritic alloys, the presence of Luders Bands that may occur on the surface of the material after deformation was examined. Lüders bands are the inability to remove the orientation effect of deformation by heat treatment after deformation of ferritic materials. This is attributed to the recovery kinetics of the ferrite phase and the industrial applicable limitations of the heat treatment time. In intensive heat treatments for complete recovery of the grains, carbides precipitated at the grain boundaries cause more problems. For this reason, mechanical tempering is more important for ferritic materials, and a smoother elasticplastic transition is aimed by the dislocation distribution mechanism of the irregular yield zone in the yield zone of annealed ferritic alloys with pronounced yielding [16,17].

Figure 6: Deep tensile test specimen according to ASTM E643 Standard

Tensile Test

In order to determine the mechanical properties of all alloys used in the experimental studies, tensile tests perpendicular to the rolling direction were applied in accordance with TS EN ISO 6892-1 Standard and the values given in Table 8 were obtained.

Table 8: Tensile Test Result

Alloy (AISI)	Thickness (mm)	Tensile Strength (Mpa)	Yield Strength (Mpa)	Elongation (%)	Hardness (Hv1) (Before)	Hardness (Hv1) (After)
441	0,39	553	344	29	166	168

Intergranular corrosion test

For the intergranular corrosion test of the alloys in accordance with ASTM A262, TS EN ISO 3651 standard, the sample was taken and immersed in the corrosion solution pool. The visual of the sample is shown in Figure 7 below. Sensitisation heat treatment was applied to determine the resistance due to the internal structure of the material against intergranular corrosion. After the corrosion test, bending test was applied to the test pieces. The test piece was bent at least 90° in a vice with a radius not exceeding the thickness of the test piece. The bent test piece was examined at low magnification (10X) for the presence of cracks. These images are shown in Figure 8.

Figure 7: Intergranular corrosion test specimen

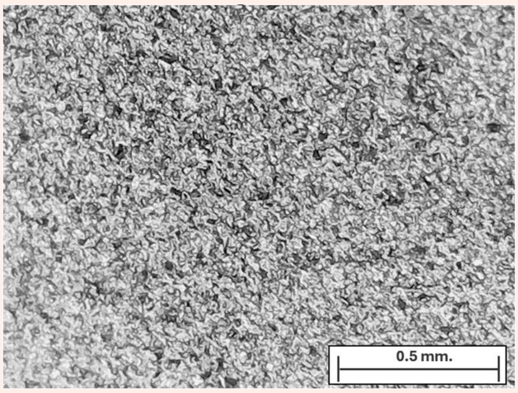

Figure 8: 10x magnification hand microscope image of 90° bent specimen after intergranular corrosion test

Metallographic studies

Samples were taken from the completed materials and metallographic preparations were made and examined under an optical microscope. Sanding was performed with SiC abrasives of 180-320-600-800-800-1000 and 1200 mesh respectively. Then, images were taken from the polished and electrolytically etched sample with V2A (200 ml. H_2O, 200 ml. HCl, 20 ml. HNO_3). These images are given in Figure 9 and Figure 10 below.

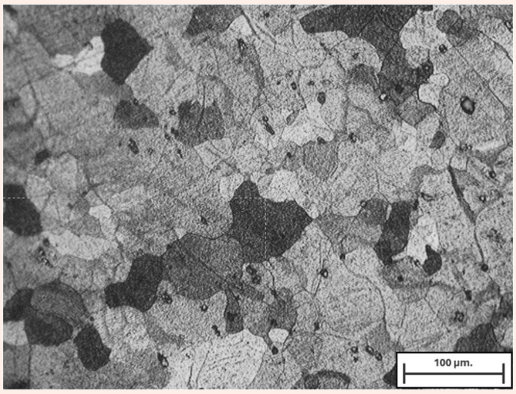

Figure 9: 100x magnified cross-sectional metallography image

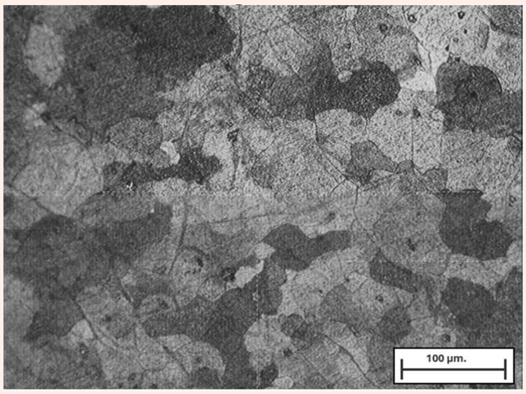

Figure 10: 100x magnified surface metallography image

Results and Discussion

AISI 441 ferritic matrix double stabilised stainless steel comes to the forefront in today's industry due to its economic and high corrosion resistance compared to ferritic matrix stainless steels despite its low nickel content. When evaluated in terms of cold rolling process, no obvious difference was observed compared to other ferritic grades. The problem of optimisation in narrow ranges in continuous heat treatment and surface treatment lines is possible with complex optimisation of line design and acid compositions. The requirement of lower concentrations of HNO_3 and HF mixed acids compared to the pickling process of austenitic stainless steels constitutes the biggest obstacle to obtaining the desired surface quality. At high acid concentrations, a pre-treatment has become mandatory in the pickling process of the alloy showing an over-pickling surface. The electro-pickling process, which stands out with its ecological solutions, is especially prominent in the production of ferritic grades that form spinel oxides. In the pickling problems experienced in the production of AISI 441, bipolar electro-pickling process was performed in the 2nd acid tank using 3000 A + 2500 A current. Nitric acid used as electrolyte increased the passive oxide layer development of AISI 441 alloy and paved the way for obtaining a clean surface. The problem in front of the optimisation of continuous heat treatment lines is that heat treatment time and pickling times increase or decrease in direct proportion. Especially with increasing deformation rates, the less heat treatment time and temperature required directly affect the pickling parameters. Difficult cleaning of high temperature oxides of ferritic grades is a known problem. For this, melt salt pre-treatment is recommended in the literature and handbooks. In the AISI 441 alloys produced within the scope of this study, melt salt pretreatment was not used for production efficiency and ecological reasons. Instead, 15% H_2SO_{4+} 15% HCL+ 1.5% H_2SO_2 by mass, which has not been used in the process before, was used. This mixture, which was used as pre-descaling, was useful for the removal of spinel oxides close to the surface and for the preparation for the pickling process.

When the mechanical properties were examined, the material could be produced in accordance with TS EN 10088-2 standard. Especially the % elongation value is much higher than the lower limit of 18% of the standard. The low elongation seen in ferritic grades causes mechanical damage in forming processes. The alloy produced is also suitable for its application areas in terms of formability. The compression-tensioning process used in the mechanical tempering process was sufficient to remove the lubricant bands. When the cup tests obtained are examined, it is seen that the obvious traces of the lubricant bands are gone. The alloy with Ti-Nb dual stabiliser maintained its homogeneous grain structure after heat treatment and no cracks etc. indicating intergranular corrosion were observed in the intergranular corrosion test.

Conclusion

- AISI 441 production, cold rolling process is similar to ferritic grades.
- It has a narrow manufacturability in terms of optimisation of heat treatment and surface treatment parameters.
- The prerequisite for an efficient production is to eliminate the molten salt process.
- Sulfuric acid, hydrochloric acid and hydrogen peroxide solution used for AISI 441 showed the desired performance in the preliminary descaling process.

- Although the mechanical properties of the alloys produced are in accordance with the relevant standard, the elongation value is higher than expected for forming processes.
- No corrosion cracks were found in the intergranular corrosion test performed on the produced alloy.

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