Microstructure and Mechanical Properties of AISI 304L Austenitic Stainless Steel Processed by Various Schedules of Rolling

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Abstract. The paper studies various rolling schedules implemented at 500°C (incl. direct, reverse, and cross rolling) and their effect on the structure formation and mechanical properties in AISI 304L stainless steel samples. Both TEM and SEM research techniques were applied. An ultrafine grain-subgrain microstructure was found to be formed inside elongated original grains. Rolling-processed microstructural elements were close in their size with the minimum value observed after a reverse rolling (240 nm). Mechanical properties were studied using microhardness measurements and tensile testing revealing a considerable increase in strength accompanied by a density reduction upon deformation. The strength values in the material subjected to all three rolling schedules are relatively close to the highest yield strength and ultimate tensile stress observed after reverse rolling with a strain degree of 70%.

1. Introduction
Corrosion-resistant steels are widely used in the industry, but their relatively moderate strength limit the area of their application. Recently, the techniques of severe plastic deformation (SPD) have been applied to induce high strain degree and enhance the strength of corrosion-resistant austenitic steels. Ultra-fine grained (UFG) microstructure with a grain size of 100-1000 nm and high-angle boundaries is processed during SPD [1-3]. This type of microstructure results both in strength and service properties enhancement with a sufficient ductility [3].

However, SPD treatment cannot be used to produce blanks. Instead of it rolling within a large temperature and strain degree range is used to fabricate UFG blanks with enhanced mechanical properties. Is is shown that cold as well as hot deformation lead to a significant refinement of grain structure and the material strengthening [4-8]. Notably, the grain refinement in austenitic steels can be attributed to deformation twinning and martensitic transformation as well [3]. The rolling temperature
decrease promotes strengthening owing to dislocation density increase and deformation twinning development [6-7].

The aim of this paper is to study the effect of rolling schedules on both the structure and properties of AISI 304 austenitic stainless steel. Direct, reverse and cross rolling at 500°C with a strain rate of 5 s⁻¹ and a relative deformation of 50 and 70% were used.

2. Experimental procedure
AISI-304L steel was used as a material for study after 1 hour holding at a temperature of 1050 °C and cooling in water, the chemical composition of which is shown in Table 1. The samples in the form of a disc with a diameter of 60 mm and a height of 15 mm were taken.

Rolling was carried out on a six-roller strip mill HANKOK M-TECH INDUSTRIES CO LTD with a roll diameter of 70 mm. The total relative deformation was 50 and 70% (which is equal to the true strain \( \varepsilon = 0.8 \) and \( \varepsilon = 1.1 \), respectively). Deformation by rolling was carried out in 15 passes, the relative reduction per pass was 5%. The workpieces were preheated in a Nabertherm N321/13 muffle furnace to 500°C before each pass. Samples for measuring microhardness were cut out transversely to the direction of sample rolling with the use of electro-erosion machine ARTA120. Mechanical tensile testing was carried out at a room temperature in accordance with GOST 1497-84, on a universal testing machine Instron 5982 at a crosshead speed of 1 mm / min.

| C      | Si   | Mn  | Ni   | Cr    | P     | S    |
|--------|------|-----|------|-------|-------|------|
| 0.024  | 0.516| 1.063| 7.64 | 17.04 | 0.022 | 0.032|

The microstructure was studied using an Olympus PME 3 optical microscope. The samples for the metallographic analysis were electrolytically etched in aquafortis at 3 V, room temperature. TEM analysis was performed at JEM-2100 transmission electron microscope operated at 200 kV. Thin foils for transmission electron microscopy (TEM) were mechanically ground to 90 µm and thinned to perforation by twinjet electrolytic polisher with a solution of 10% HClO₄ in CH₃COOH at 25 V.

3. Results and discussion

3.1. The microstructure of AISI-304L austenitic stainless steel.
The AISI-304L austenitic stainless steel has a grain size of 25 µm upon water quenching from 1050°C (holding for 1 hour). After processing at a strain degree of 70%, metallographic specimens were cut from a lateral side of deformed billets along the direction of a rolling plane (figure 1). A strongly oriented grain microstructure can be observed after all three rolling schedules: with original structure elongated along a deformed sample (figure 1a,b) after direct and reverse rolling with a strain degree of 70%, and elongated at 60° to a deformed sample after a cross rolling (figure 1c).
About 100-150 measurements in increments of 200µm were taken to estimate the distance in the transverse direction of elongated grains. The material has nearly the same mean transverse grain size following all the three rolling schedules with a strain degree of 70%: 8.6µm for direct rolling, 8.4µm for reverse rolling and 8.7µm for cross rolling. A strong orientation of original grains and their similar transverse dimensions seem to be attributed to the absence of discontinuous dynamic recrystallization (by Bailey and Hirsch mechanism) during various rolling schedules. Only TEM studies are capable of providing the insight in the development of a continuous dynamic recrystallization (by the Cahn-Burger's mechanism) in the material. Microscopic analysis was used to investigate foils in the specimens' plane similar to a rolling direction. Microstructural elements were measured along a rolling plane (i.e. grain/subgrain transverse size). In spite of the difference in rolling schedules, a fine microstructure has almost the same size of structural elements and dislocation density. At the same time, the highest thickness and density in deformation bands were found after a direct rolling, and the most homogeneous microstructure observed after a cross rolling.

A direct rolling resulted in the formation of an inhomogeneous microstructure. Its shear bands or microbands contain strongly elongated thin grains/subgrain with large misorientations. The size of grains/subgrains is about 260 nm in the cross section and 100 nm in bands. Dislocation density inside grains/subgrains is $9 \times 10^{14}$ m$^{-2}$. According to TEM (figure 2), the processed material has a grain/subgrain microstructure with the development of continuous dynamic recrystallization.

A reverse rolling leads to the formation of a more homogeneous microstructure with slight misorientations of subboundaries. However, some shear bands similar to microbands with large misorientations are observed. Grains/subgrains have the size of about 240 nm with dislocation density of $10 \times 10^{14}$ m. TEM reveals the development of a partial continuous dynamic recrystallization as well.

Cross rolling results in the processing of a homogeneous fine microstructure with slight misorientations of subboundaries. Shear microbands are rarely found. Grains/subgrains in the cross direction have a size of 250 nm with a dislocation density inside grains/subgrains of $7 \times 10^{14}$ m$^{-2}$.

The development of partial continuous dynamic recrystallization is also proved by TEM.
3.2. Study of mechanical properties.
The mechanical properties after tensile testing of all the samples subjected to various treatment types are shown in figure 3. The data obtained reveal the increase in a yield strength and ultimate tensile stress after rolling with higher values achieved by applying a high strain degree of 70%. The highest ultimate tensile stress of 972 MPa and yield strength of 938 MPa at a strain degree of 70% are found in the
material subjected to a reverse rolling. And the lowest ultimate tensile stress of 937 MPa and yield strength of 911 MPa are observed in the material after cross rolling. However, the mechanical characteristics processed after various types of rolling are quite similar to each other. Somewhat larger strength values in the material after a reverse rolling with a strain degree of 70% are possible caused by slightly smaller structural elements. Ductility in the specimens after rolling decreases from 52% after thermal treatment to 28-34% after a strain degree of 50% and to 11-22% at a strain degree of 70%. The highest ductility is observed after a cross rolling, with 34% at a strain degree of 50% and with 22% at a strain degree of 70%.

Microhardness HV was measured in the central region of a cross section of a specimen subjected to three rolling schedules. The results are shown in Fig. 4, and the analysis reveals that microhardness is distributed in a rather inhomogeneous nature along the section. Microhardness along the specimen's section differs by 50-100 HV with the highest value of 350-450HV observed after a cross rolling.

Figure 3. Mechanical properties after tensile testing of the samples subjected to direct, reverse and cross rolling

Figure 4. Change in the microhardness of the cross section of the samples after direct (a) reverse (b) and cross rolling (c) when reaching a maximum deformation of 70%
Direct and reverse rolling results in an inhomogeneous distribution of microhardness values along the section. Higher strength of up to 320HV is observed in surface layers of the specimen subjected to a direct rolling, while a middle layer has a strength of ~60HV less. A reverse rolling leads to a more homogeneous distribution of microhardness with the difference between a middle and a surface layer of ~20HV. The highest microhardness of 400±40HV is observed in specimens processed by cross rolling.

4. Conclusions
1. The effect of rolling schedules (direct, reverse, cross rolling) at 500°C and a relative strain degree of 50 and 70% on the microstructure formation and the mechanical properties of AISI 304L stainless austenitic steel is studied.
2. Both metallographic and TEM analysis of AISI 304L microstructure subjected to the three rolling schedules with a strain degree of 70% were performed. Grain-subgrain microstructure with structural elements of 240-260 nm is processed in strongly oriented original grains. The development of a continuous dynamic recrystallization with the formation of grains with high-angle boundaries and subgrains with low-angle boundaries is confirmed by TEM.
3. Rolling results in the growth of both yield strength and ultimate tensile stress with higher values achieved at a strain degree of 70%. The increase in a strain degree from 50 to 70% enables to enhance mechanical properties by around 12% as compared to the 30% of an initial state subjected to thermal treatment. The highest ultimate tensile stress of 972 MPa and yield strength of 938 MPa at a strain degree of 70% were observed after reverse rolling with the lowest values of 937 MPa and 911 MPa respectively were achieved after a cross rolling. However, the characteristics processed after various types of rolling are quite similar to each other.

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