An analysis of microstructure and mechanical properties of ferritic stainless steel 430 during cold rolling and subsequent annealing

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Abstract
Systematic study on the microstructural evolution and mechanical properties of ferritic stainless steel (FSS) 430 with different annealing processes was carried out in the present work. The results show that microstructural refinement can be achieved by optimization of annealing processes, improving elongation, yield strength, and tensile strength of FSS 430. An optimal annealing temperature of 950 °C is found with better homogeneity and enhanced mechanical properties among the cold-rolled and annealed FSSs. During annealing processes, the fraction of high-angle grain boundaries of FSS 430 annealed at 950 °C is found to be the highest, indicating that a homogeneous microstructure with high recrystallization rate is formed inside the FSS 430 strips. In addition, high fraction of the hard grains (SF < 0.4) and low fraction of the soft grains are found inside FSS 430 annealed at 950 °C, improving the plasticity of material. Overall, the optimization of annealing processes benefits the microstructural refinement of FSS and thereby improving the mechanical properties of materials.

Keywords Annealing · Microstructure · Mechanical properties · Ferritic stainless steel

1 Introduction
Owing to high corrosion resistance, excellent mechanical properties and low cost, ferritic stainless steel is extensively utilized in a variety of fields such as home alliance [1–3], MEMS systems [4, 5], medical science [6–8], and vehicle engineering [9, 10]. Nevertheless, the usage of FSS in industrial fields is still limited by the formability and mechanical properties compared to its counterparts (austenitic stainless steel, for example). In order to improve the mechanical properties of FSS, the application of heat treatment during rolling processes is commonly utilized to enhance the mechanical properties of steels through microstructural refinement induced by optimization of rolling processes [11–13].

Extensive research has been carried out to enhance the mechanical properties of steels via optimization of rolling processes. Meng et al. [14] systematically studied the mechanical properties of FSS with different quenching and partitioning processes, and an AISI 430 with both high strength and good plasticity was achieved by a recrystallization annealing before quenching and partitioning process. The variation of mechanical properties of cold-rolled FSS 430 during continuous annealing process was investigated by Zhang et al. [15], and the effects of annealing temperature on the strength, elongation, and anisotropy properties of FSS 430 were analyzed based on a series of rolling tests. The effects of intercritical annealing process on microstructure and tensile properties of cold-rolled 7Mn steel were investigated by Yang et al. [16]. It is found that the tensile properties of 7Mn steel is significantly affected by both intercritical annealing temperature and duration. Li et al. [17] assessed the effect of rolling on tensile properties of austenitic steel, and found that the well balance between the κ-carbides precipitation and the dislocation recovery
contributes to the optimized tensile properties after rolling at a reduction of 40%. A series of rolling tests were performed by Li et al. [18] to evaluate the effect of finish rolling temperature on the mechanical properties of tempered steel. It is found that the yield and tensile strength of the steels were increased firstly and then decreased along with the decrease of finish rolling temperature. Sahoo et al. [19] studied the deformation behavior in medium-Mn steel processed through intercritical/cold rolling and annealing processes, and found that higher yield stress and ultimate tensile strength can be achieved through intercritical rolling processes. The effects of cold rolling prior to the intercritical heat treatment on mechanical properties of 4340 steel was analyzed by Hosseinifar and Ekrami [20]. It is found that the improvement of mechanical properties of 4340 steel were related to increase in martensite volume fraction and ferrite grain refinement. In order to investigate the relationship between microstructure and mechanical properties of steels, Lemarquis et al. [21] studied the cold rolling effects on the microstructure properties of 316L stainless steel, and found that the difference of initial microstructure regarding the grain size distribution and texture structure affects the extent of grain refinement and texture decay. The microstructural characterization of FSSs during recrystallization annealing were analyzed by Tanure et al. [22] through tensile tests and Swift tests, and the severity of earing of FSS sheets were found to be dominated by the homogeneity of the γ-fibre distribution. Cai et al. [23] evaluated the influence of annealing temperature on microstructure and tensile properties of cold-rolled Fe-0.2C-11Mn-6Al steel, and found that the refinement of microstructure contributes to the enhancement of tensile properties. The effect of grain size and initial texture on microstructure and formability of cold-rolled FSS was analyzed by Rodrigues et al. [24], and excellent formability of FSS was achieved with microstructure comprising a strong γ fiber and a weak θ fiber after two-step cold rolling.

Above all, it is commonly acknowledged that the mechanical properties of steel can be improved through microstructural refinement induced by optimization of rolling and subsequent annealing processes [22–24]. Nevertheless, the microstructural evolution of FSS during rolling processes is still not clear. Additionally, the effect of rolling processes on the enhancement of mechanical properties of FSS remains to be explored in order to obtain rolled products with high strength and good plasticity. The objective of the present study is to evaluate the relationship between microstructure and mechanical properties of FSS 430, and thereby improving the mechanical properties of FSS 430 through optimization of rolling and subsequent processes. In this manuscript, the microstructural evolution and mechanical behavior of FSS 430 will be systematically explored based on a series of experiments, aiming to improve the mechanical properties.

### Table 1 Chemical compositions of FSS 430 (wt%)

| Element | C | Si | Mn | S  | P  | Cr  | N  |
|---------|---|----|----|----|----|-----|----|
| C       | 0.048 | 0.34 | 0.24 | 0.002 | 0.032 | 16.25 | 0.035 |

![Fig. 1 Schematic diagram of rolling and annealing processes used in the present work](image)
of the rolled FSS strips through optimization of rolling and subsequent annealing processes.

2 Materials and experimental procedures

The material used in the present work was a 5-mm-thick FSS 430 strip, and its chemical compositions are given in Table 1. Figure 1 shows the schematic diagram of the whole experimental process, which consists of cold rolling and annealing processes. In the present work, the as-received ingot was cold-rolled with different reductions ranging from 55 to 75% in order to evaluate the effect of reductions on the microstructural evolution and mechanical properties of FSS. After cold rolling experiments, heat treatment processes with different annealing temperatures (800 °C for 5 min, 850 °C for 5 min, 900 °C for 5 min, 950 °C for 5 min, and 1000 °C for 5 min) were applied to obtained specimens.

After rolling experiments, the specimens were cut and polished with sandpaper and then electrolytically polished for further observation. The specimens were characterized using scanning electron microscopy (SEM) with electron backscatter diffraction (EBSD). In this work, the tests were carried out with a step size of 0.75 μm. To investigate the microstructural evolution of specimens with different rolling processes, the crystallographic orientations of grains inside FSS were obtained and analyzed on the basis of the orientation distribution functions (ODFs). It is known that FSS has a body-centered cubic (BCC) crystal structure, the microstructural evolution of FSS strips were analyzed based on ODFs inside the ϕ2 = 45° section of the Euler space [25].

| Annealing temperature (°C) | As-received | 800 | 850 | 900 | 950 | 1000 |
|---------------------------|------------|-----|-----|-----|-----|------|
| Average grain size (μm)   | 5.02       | 7.95| 6.97| 7.33| 8.71| 10.82|

Fig. 2 ND-IPF of cold-rolled and annealed FSS 430 specimens with different annealing temperatures of (a) untreated, (b) 800 °C, (c) 850 °C, (d) 900 °C, (e) 950 °C, and (f) 1000 °C
On the other hand, a series of tensile tests were conducted at room temperature with the stroke speed of 1 mm/min. The objective of tensile tests is to investigate the variation in properties of the material. Figure 3 shows the grain size distributions of FSS 430 specimens with different annealing temperatures. Figure 4 presents the ODFs of the FSS 430 specimens with various annealing temperatures, along with the location of ideal orientations and fibers.
of the mechanical properties of FSS with different rolling processes. The obtained specimens were cut into tensile samples based on the ASTM E8-E8M standard [26] along the rolling direction (RD). Each group of tensile tests was performed for three times in order to reduce the errors generated during experiments.

3 Results

3.1 Microstructure

Figure 2 shows the microstructure of the cold-rolled and annealed FSS 430 specimens with different annealing temperatures. The obtained inverse pole figures (IPF) coincides with the center of the FSS specimens. It is seen that the as-received FSS specimen was dominated by elongated grains (as shown in Fig. 2(a)) while the ferrite grains in cold-rolled and annealed specimens were mainly recrystallized, equiaxed grains (as shown in Fig. 2(b-f)). After cold rolling processes, an inhomogeneous microstructure was formed, which can be attributed to the work hardening difference between grains with different orientations [27]. During annealing processes, a number of nucleation sites were generated from high energy sites (for example, the deformation bands), leading to the formation of new grains with different orientations. Consequently, remarkable weakening of the {110} <110> and the brass components occurs during annealing processes. The specimen annealed at 800 ºC still exhibits a strong brass texture and the intensity is further weakened with the annealing temperature increases to 850 ºC. With the annealing temperature increases from 850 to 950 ºC, a homogeneous and uniform microstructure is formed inside FSS and remarkable weakening of the {110} <110> and the brass components can be observed, as shown in Fig. 4e. With the annealing temperature further increases from 950 to 1000 ºC, an increase of the intensity of the {110} <hkl> components is found, which can be attributed to the formation of coarse bands and inhomogeneity of FSS.

Additionally, it is worth mentioned that remarkable reduction of the fraction of the {112} <110> component is observed inside FSS 430 annealed at 950 ºC, which can be attributed to the grain refinement during annealing processes. It is known that the coarse grain bands inside the annealed FSS is dominated by the {112} <110> component and components along the {110} <hkl> fibre with the maximum intensity of 20 (as shown in Fig. 4a). Given that the microstructure of the as-received FSS 430 is dominated by the elongated grains, the elongated grains inside the as-received FSS 430 are mainly grains with the {110} <110> and the brass components (as shown in Fig. 5). During annealing processes, a number of nucleation sites were generated from high energy sites (for example, the deformation bands), leading to the formation of new grains with different orientations. Consequently, remarkable weakening of the {110} <110> and the brass components occurs during annealing processes.

![Figure 5 Volume fraction of major texture components of FSS annealed at different temperatures](image)

| Annealing temperature (ºC) | As-received | 800 | 850 | 900 | 950 | 1000 |
|----------------------------|-------------|-----|-----|-----|-----|------|
| Recrystallized fraction (%) | 0.7         | 66.1| 68.5| 75.0| 81.9| 88.8 |
Fig. 6 Grain orientation spread map of FSS 430 specimens with different annealing temperatures of (a) untreated, (b) 800 °C, (c) 850 °C, (d) 900 °C, (e) 950 °C, and (f) 1000 °C.
The reduction of the \{112\} \textless 110\textgreater components indicate that fewer coarse grain bands were formed inside FSS 430 strips annealed at 950 \(^\circ\)C. With the increase in annealing temperature from 950 to 1000 \(^\circ\)C, obvious increase in the fraction of the \{112\} \textless 110\textgreater component is observed, indicating that a number of coarse grain bands were formed inside FSS annealed at 1000 \(^\circ\)C (as shown in Figs. 4 and 5).

Figure 6 shows the grain orientation spread (GOS) maps of FSS 430 annealed at different temperatures, and the recrystallized fraction is depicted in Table 3. In general, the regions with GOS \(< 1^\circ\) are considered to be recrystallized [31]. With the increase in annealing temperature from 800 to 1000 \(^\circ\)C, the recrystallized fraction gradually increases from 66.1 to 88.8%. For specimens annealed at 800 to 950 \(^\circ\)C, the increased fraction of recrystallized grains promotes the generation of equiaxed grains, leading to remarkable microstructural refinement and thereby improving the mechanical properties of FSS 430. While for specimens annealed at 950 to 1000 \(^\circ\)C, the growth of grains promotes the formation of coarse grain bands and enhances inhomogeneity of microstructure, reducing strength and plasticity of FSS. The control of recrystallized fraction via annealing, therefore, is critical to the refinement of microstructure. It is known that the recrystallized fraction is significantly affected by the dislocation density of specimens, the dislocation density of FSS 430 specimens were analyzed, as shown in Table 4.

Figure 7 shows the calculated geometrically necessary dislocation (GND) density distributions in the FSS 430 specimens annealed at different temperatures, which reflects the local changes in dislocation density and their distribution in the microstructure of FSS 430 [13]. It is seen that the as-received FSS 430 is dominated by grains with very low dislocation density. With the increase in annealing temperature from 800 to 1000 \(^\circ\)C, a decrease in dislocation density is observed. Considering that the strain strengthening induced by high dislocation density contributes to the increase in the material strength, the variation of mechanical properties of FSS can be estimated through the dislocation density of materials [32, 33].

### Table 4 Volume fractions of LAGBs and HAGBs of FSS 430 with different annealing temperatures

| Annealing temperature (°C) | As-received | 800 | 850 | 900 | 950 | 1000 |
|----------------------------|-------------|-----|-----|-----|-----|------|
| Volume fraction of LAGBs (%)| 74.9        | 8.8 | 9.0 | 8.7 | 6.3 | 8.5  |
| Volume fraction of HAGBs (%)| 22.1        | 88.3| 87.9| 88.8| 91.0| 88.3 |

#### 3.2 Grain boundaries

Figure 8 shows the misorientation distributions of FSS 430 annealed at different temperatures. For the as-received FSS specimens, the ferrite grains are dominated by the low angle grain boundaries (LAGBs, 2° < misorientation angles < 15°) due to the formation of subgrains during cold rolling [34]. The existed high angle grain boundaries (HAGBs, misorientation angles > 15°) are mainly originating from the accumulation of dislocations. For FSS annealed at 800 °C, remarkable reduction of the LAGBs can be observed, indicating that active recovery and recrystallization occurs during annealing processes. Additionally, the fraction of the LAGBs decreases with the increase in annealing temperature from 800 to 950 °C, following by an increase with the annealing temperature further increases from 950 to 1000 °C. Considering the formation of the HAGBs is mainly induced by the arrangement of dislocations and recrystallization [35], the fraction of HAGBs of FSS 430 annealed at 950 °C is found to be the highest (as shown in Table 4), indicating that a homogeneous microstructure with high recrystallization rate is formed inside FSS. The annealing temperature of 950 °C, therefore, is considered to be optimal to microstructural refinement of FSS 430.

In order to study the formation of coarse grain bands of FSS 430 annealed at different temperatures, the degree of coincidence at ferrite grain boundaries were explored. Figure 9 shows the coincidence site lattice (CSL) boundaries of FSS 430 specimens annealed at different temperatures. It is seen that the volume fraction of the CSL boundaries inside the as-received
Fig. 9 CSL boundaries of FSS 430 specimens with different annealing temperatures of (a) untreated, (b) 800 °C, (c) 850 °C, (d) 900 °C, (e) 950 °C, and (f) 1000 °C
FSS 430 is 3.86%, and it was dramatically increases to 16.3% with the increase in annealing temperatures to 1000 °C. Considering the grain boundary mobility can be affected by the fraction of CSL boundaries, the fraction of low mobility ($\Sigma 3$) boundaries and high mobility boundaries ($\Sigma 5, \Sigma 7, \Sigma 9$) [36, 37] are analyzed, as shown in Table 5. With the increase in annealing temperature from 800 to 1000 °C, the fraction of low mobility boundaries remarkably increased from 1.77 to 2.95% and the high mobility boundaries slightly increased from 2.39 to 2.86%, followed by a decrease to 2.46%. In this work, the fraction of the high mobility CSL boundaries remained within close range with the increase in annealing temperature from 800 to 1000 °C, indicating that the variation of grain boundary energy is negligible for the annealed specimens in the present work. Considering the required driving force for grain growth is proportional to the grain boundary energy and inversely proportional to the grain size, the effect of grain boundary energy can be ignored and the grain boundary mobility depended on the grain size. For specimens annealed at 800 to 950 °C, sufficient thermal activation for the grain growth of refined grains are provided, resulting in the formation of a homogeneous microstructure with few coarse grain bands. With the further increase in annealing temperature to 1000 °C, the heat treatment contributes sufficient thermal activation and accelerates the grain growth kinetics, leading to the increase in the grain size of FSS 430.

During rolling processes, energy is introduced and the total stored energy of the material increased. To reach the minimum energy configuration, the LAGBs grows at the expense of the HAGBs. Grain rotation, accompanying the rolling and annealing process, providing the driving energy for the formation of $\Sigma 3$, leading to an increase in the fraction of $\Sigma 3$ during rolling and annealing process. It is known that $\Sigma 3$ is a low energy and low mobility boundary type defined by a rotation of 60° around a common axis $<111>$, the grain bands with planes and directions such as (111) [1 $\bar{1}$ 0]/(111) [0 $\bar{1}$ 1] and (111) [1 $\bar{1}$ 1]/(111) [1 1 2] tend to form with the increase in the fraction of $\Sigma 3$, thereby affecting the microstructural homogeneity of materials. On the other hand, the high mobility boundaries are likely to be formed combining the with the formation of the Goss component [38], thereby affecting the homogeneity of materials.

### 3.3 Mechanical properties

Figure 10 shows the stress–strain curves of FSS 430 specimens with different annealing temperatures, and the mechanical properties of FSS specimens were displayed in Fig. 11. It is noted that for the cold rolled and annealed FSS 430 specimens, the elongation (EL) gradually increases from 30.7 to 33.6% along with the annealing temperature increases from 800 to 950 °C, following by a dramatic decrease to 31.7% with the annealing temperature further increases to 1000 °C. With the increase in annealing temperature from 800 to 950 °C, both the ultimate tensile strength (UTS) and yield strength (YS) show similar trend and slightly increases to the peak value of 450.7 MPa and

| Annealing temperature (°C) | As-received | 800 | 850 | 900 | 950 | 1000 |
|---------------------------|-------------|-----|-----|-----|-----|------|
| Volume fraction of CSL boundaries (%) | 3.86 | 13.40 | 13.71 | 14.56 | 16.30 | 16.65 |
| Low mobility ($\Sigma 3$) boundaries (%) | 0.64 | 1.77 | 1.82 | 2.32 | 2.48 | 2.95 |
| High mobility ($\Sigma 5, \Sigma 7, \Sigma 9$) boundaries (%) | 0.50 | 2.39 | 2.49 | 2.83 | 2.86 | 2.46 |

Fig. 10 Stress–strain curves of FSS 430 specimens with different annealing temperatures

Fig. 11 Elongation and tensile strength of FSS 430 specimens with different annealing temperatures
Fig. 12 Schmid factor of FSS 430 specimens with different annealing temperatures of (a) untreated, (b) 800 °C, (c) 850 °C, (d) 900 °C, (e) 950 °C, and (f) 1000 °C.
256.5 MPa, respectively. It is known that the reduction of average grain size enhances the YS based on Hall-Patch effect [31], the microstructural refinement induced by optimization of annealing processes, therefore, contributes to the increase in UTS and YS. Instead, once the annealing temperature further increases to 1000 °C, both the UTS and YS decrease sharply to 443.0 MPa and 189.0 MPa, respectively due to the formation of course grain bands inside FSS 430. Among FSS 430 specimens annealed at different temperatures, the specimens annealed at 950 °C show the best mechanical properties with the highest elongation and strength, indicating that an optimal annealing temperature of 950 °C is obtained for the improvement of the mechanical properties of FSS 430.

3.4 Twinning characteristic in tension of FSS

Twinning is one of the major deformation mechanisms of FSS 430 [39]. The activation of twinning leads to yielding phenomenon [40] and affects the strain hardening behavior of materials [41–43]. In order to investigate the microstructural evolution and mechanical properties of FSS 430 annealed at different temperatures, the Schmid factor (SF) maps of the annealed FSS strips were analyzed, as shown in Fig. 12. It is seen that the SFs of most twins beyond 0.4. In general, the high SFs indicates that twinning are easy to activate and thereby coordinating further deformation, whilst low SFs indicates the grains rotated to orientations that are difficult to activate. In order to further evaluate the fraction of low SF (<0.4) twins, the distribution of SF of twins are analyzed (as shown in Fig. 13). It is seen that the fraction of low Schmid factor twins of FSS 430 strips annealed at 950 °C is the highest among annealed FSS strips, indicating that few grains are soft oriented which contributes to grain boundary migration [44]. The soft grains with a high SF are prone to slippage, causing low yield strength of materials. On the other hand, the hard grains with a low SF are difficult to activate the slip system, promoting the generation of twinning to coordinate further deformation [29]. It is seen that the fraction of the soft grains (0.5 > SF > 0.45) are gradually reduced with the increase in annealing temperature from 850 to 1000 °C, indicating that twinning would be increasingly difficult to activate.

4 Conclusions

In summary, the microstructure and mechanical properties of the cold-rolled and annealed FSS strips were analyzed through a series of tests, and the conclusions are drawn as follows:

1 Microstructural refinement can be achieved by optimization of annealing processes, improving the elongation and tensile strength of FSS 430. An optimal annealing temperature of 950 °C is found with better homogeneity and enhanced mechanical properties of FSS 430.

2 The fraction of the HAGBs of FSS 430 annealed at 950 °C is found to be the highest, suggesting that a homogeneous microstructure with high recrystallization rate is formed inside FSS 430 annealed at 950 °C. In addition, the fraction of the CSL boundaries increases with the increase in annealing temperatures, while the fraction of the high mobility reaches the peak value in FSS 430 annealed at 950 °C, leading to the refined, homogeneous bands inside FSS.

3 FSS 430 specimens annealed at 950 °C have the highest tensile strength, elongation value and the yield strength among all tested specimens. The improved plasticity of material is mainly attributed to the microstructural refinement of FSS 430 induced by optimization of annealing processes.

4 The fraction of the low Schmid factor twins of FSS 430 annealed at 950 °C is the lowest among specimens. This means that most grains inside FSS 430 annealed at 950 °C are soft oriented grains that are easy to activate, improving the plasticity of material.

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Data availability  All data gathered regarding this publication is presented.

Code availability  Not applicable.

Declarations

Ethics approval  All procedures performed in studies involving human participants were in accordance with the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki declaration and its later amendments or comparable ethical standards.

Consent to participate  Informed consent was obtained from all individual participants included in the study.

Consent for publication  The participant has consented to the submission of the case report to the journal.

Conflict of interest  The authors declare no competing interests.

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