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Effect of drawing and annealing on the microstructure and mechanical properties of 304 austenitic stainless steel wire

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Abstract

Plastic deformation at room temperature, and the proceeding heat treatments, are important processes for optimizing the microstructure and mechanical properties of austenitic stainless steel. The microstructure and mechanical properties of cold-drawn 304 austenitic stainless steel wire were investigated after annealing at 700 °C and 800 °C, with different times (20, 40 and 60 min) and drawing strain (0.4, 1.0 and 1.5). Electron backscattered diffraction (EBSD) techniques, transmission electron microscope (TEM) analysis, differential scanning calorimeter (DSC) and tensile tests were performed in order to study the microstructure evolution and mechanical properties during different annealing processes for the 304 austenitic stainless steel wire. The results showed that the quantity of \( \alpha’ \) martensite and dislocations increased with an increase in the strain, which means that, while the ultimate tensile strength of the cold-drawn wires elevated, the elongation reduced. The mechanical properties of stainless steel wires also varied with the evolution of martensite transformation characteristics, density of stacking fault, dislocation and twin, as well as the recrystallization degree under various annealing conditions. The recrystallization temperature of steel wire was mainly determined by the magnitude of the strain, while the martensite reversal temperature was determined by the stacking fault energy and the deformation value. The temperature of recrystallization and martensite reverse in steel wire decreased with the increment of the strain. The balance of tensile strength and elongation of steel wire can be obtained by adopting the proper annealing process combined with cold-drawing deformation. In this paper, we showed that a good combination of strength and elongation in 304 austenitic stainless steel can be obtained with a strain of 1.5 annealed at 800 °C for 20 min.

1. Introduction

Compared with ordinary carbon steel, 304 austenitic stainless steel is widely used in the fields of transportation, aerospace and the chemical industry because of its excellent rust resistance, corrosion resistance and mechanical properties [1, 2]. In 304 stainless steel, martensite transformation mainly occurs by a low-temperature plastic deformation process, resulting in its high strength, low ductility, and non-monotonic work-hardening rate [3, 4]. The deformation-induced martensitic transformation and the reversion of deformation induced martensite to fine-grained austenite has been found to be an efficient method to increase significantly the yield strength of metastable austenitic stainless steels without impairing much their ductility [5–7]. In the last ten years, many researchers have studied the mechanism and influencing factors of martensite transformation in 304 stainless steel, and explored and discussed the influence of chemical elements, strain rate, stress state and deformation temperature on the martensite transformation process [8, 9]. Microstructural evolutions during annealing of a plastically deformed AISI304 stainless steel were investigated [10]. It was shown that the primary recrystallization of the retained austenite postpones the formation of an equiaxed microstructure, which
coincides with the coarsening of the very fine reversed grain. An advanced thermomechanical process based on the formation and reversion of deformation-induced martensite was used to refine the grain size and enhance the hardness of an AISI 304L austenitic stainless steel [11]. The tensile fracture behavior of a 304L stainless steel with heterogeneous lamella structure (HLS) is studied by Y Zhao. It is proposed that the fracture initiation originated from boundaries between ultra-fine grains (UFGs) and recrystallized micro-grains (Micro-RGs) in the HLS steel while that is from some dislocation migration barriers in the HS steel [12]. Cold-drawn austenitic stainless steel wire is now used to make high strength spring steel wire [13] but, unlike stretching and rolling deformation, severe work-hardening behavior leads to wire breakage, which often occurs in the process of wire drawing, greatly affecting production efficiency and safety. More studies have shown that the inhomogeneous martensite and twinning generated by strain-induced transformation are the fundamental cause of fractures in austenitic stainless steel wire during cold-drawing deformation [14]. Therefore, based on the widely used multipass drawing-annealing process in industrial manufacturing, the selection of parameters such as annealing temperature and annealing time between passes, and their influence on the plastic recovery of drawn stainless steel wire, are still key scientific issues that must be solved expeditiously. Moreover, the content of martensite and its distribution characteristics have a significant effect on the ductility and toughness of steel wire. Patel et al [15] and Murr et al [16] believed that the volume fraction of martensite would increase significantly under tensile and multiaxial stress states. Angel et al [17, 18] found that the martensite volume fraction increased with an increase in the strain but decreased with an increase in annealing temperature. Therefore, it is of great theoretical and engineering significance to study the microstructure evolution of 304 austenitic stainless steel in the annealing process after drawing.

In this study, a combination of advanced characterization methods, including electron backscatter diffraction (EBSD), transmission electron microscopy (TEM), transmission Kikuchi diffraction (TKD), differential scanning calorimeter (DSC) and x-ray diffraction, were selected to investigate the deformation and annealing microstructure evolution of cold-drawn 304 austenitic stainless steel wire in the drawing strain range of 0.4 to 1.5 under different annealing processes. Research work on the influence mechanism of multiple annealing factors between intermediate cold-drawing passes on the microstructure and the properties of stainless steel wire will benefit from obtaining a good match of ultimate tensile strength (UTS) and ductility in a series of industrial products.

2. Materials and methods

The chemical composition of the 304 austenitic stainless steel wire used in this experiment is shown in table 1, and the flow chart of the sample preparation is given in figure 1. Stainless steel wires with drawing strain variables of 0.4, 1.0 and 1.5 (Reduction of area 33%, 65% and 77%, respectively) were made by continuously drawing at room temperature from a wire rod with an initial diameter of 5.6 mm, and then annealed at 700 °C and 800 °C for 20 min, 40 min and 60 min, respectively, followed by air cooled. The annealed samples were cut along the longitudinal section, which were ground and electro-polished in a 10% perchloric acid alcohol solution. A field emission scanning electron microscope (SEM, TESCAN MIRA 3 XMU, operated at 20 kV) equipped with electron backscatter diffraction (EBSD) system; transmission electron microscope (TEM, FEITECNAI G2 F20, operated at 200 kV) equipped with energy dispersive spectroscopy (EDS); and x-ray diffraction (XRD, RIGAKU DMAX-2500PC, operated at 18 kW) were used to analyze the microstructural evolution of the steel wires during drawing and annealing. Tensile strength was tested under China standard of GB/T228.1 using a computer-controlled electronic universal testing machine (CMT4304) with a stretching speed of 25 mm min \(^{-1}\) and gauge length of 100 mm (Electronic extensometer model: YYU-25/50). Differential scanning calorimetry (DSC) was performed on the cold-drawn wires after surface polishing. Argon was used to protect the samples during the test, and the test temperature was raised from room temperature to 1000 °C at a heating rate of 10 °C min \(^{-1}\).

3. Results

3.1. Mechanical properties and microstructure before annealing

The UTS and elongation of the 304 stainless steel wire samples and the volume fraction of the α’ martensite in the microstructure before heat treatment are shown in figure 2. Five tensile tests were carried out for each sample.

| C  | Si  | Mn  | P   | S   | Cr  | Ni | Mo | Cu | N   | Fe   |
|----|-----|-----|-----|-----|-----|----|----|----|-----|------|
| 0.049 | 0.362 | 0.78 | 0.03 | 0.002 | 18.19 | 8.09 | 0.01 | 0.31 | 0.057 | Bal. |

Table 1. Chemical composition of the 304 stainless steel (mass fraction, %).
to obtain the average value. As can be seen from figure 2(a), the tensile strength increased from the initial 659MPa to 1551MPa, while the elongation decreased sharply from 45% to 1.0%. As the strain increased to 0.4, 1.0 and 1.5, the fraction of the α′ martensite increased, and the fraction of twin increased firstly and then decreased, as shown in figure 2(b). The contents of twin and martensite, obtained via EBSD and XRD result calculations [19].

The microstructure of the steel rod and drawing wires before annealing, including the inverse phase figures (IPF) from the EBSD or TKD and TEM results, are shown in figure 3. As can be seen from IPF maps (a-d), the equiaxed grains elongated as the strain increased. The fraction of α′ martensite increased, and the twin first increased (from strain 0.4 to 1.0) and then decreased (from strain 1.0 to 1.5), as the strain also increased figure 2(b) and figures 3(f)–(h). Once a twin is formed by dislocation interactions, it may then thicken by overlapping of other nuclei formed on the (1 1 1) planes parallel to its habit plane [20]. This is the reason why the density of twins increase with increasing strain. Bracke et al [21] have suggested that the retransformation dislocations, namely Shockley partials with a burgers vector opposite to that of the twinning dislocation, should occur during the process of intersection. The movement of these Shockley partials will eliminate twins, resulting in the reduced density of twins with increasing strain. This hypothesis provides a reasonable explanation for the present observation of abrupt drop in twin density when the strain level is greater than 1.0. The microstructure of the annealed rod consisted of equiaxed austenite grains with a large amount of α′ martensite and annealing twins (figures 3(a) and (e)). There is a certain amount of martensite in the sample with zero strain value (figures 2(b) and (e)), which can be attributed to the stability of austenite in the material and the potential carbide due to water cooling after cold rolling and annealing of wire rod. It has been shown that the presence of carbon can lead to the rise of Ms temperature in local zones around carbide particles that are depleted from alloying elements. This can lead to the local destabilization of the austenite and the formation of some amount of martensite during cooling to room temperature [22]. From the results of the TEM, the grains were refined and
elongated, the fraction of the $\alpha'$ martensite increased noticeably, and the morphology of the interface between the $\alpha'$ martensite and the austenite matrix became blurred with the increase in strain due to a high dislocation density and interfacial stress. The density of dislocations in the cold drawn samples increased with an increase in the strain. Meanwhile, the martensite in the austenite matrix was refined and transformed from strip to needle, as shown in figures 3(i)–(l). The residual lath martensite of the sample with strain 0 after hot rolling and water cooling can be observed from figure 3(i). As the strain increases to 0.4 the lath martensite is elongated, while after 1.5 the lath martensite morphology is similar to needle shape (figure 3(l)) which generated via drawing deformation rather than annealing process (figure 3(i)). The $\alpha'$ martensite can enhance the work-hardening rate.
when the amount of martensite and the number of dislocations increase with the increase in the strain, which means the strength of the cold-drawn wires elevates, but the elongation reduces.

### 3.2. Microstructural evolution during annealing

The microstructures of the cold-drawn stainless steel wire annealed at 700 °C for different times are shown in figure 4. It can be seen that the average grain size and grain boundary density of the 33% deformation samples were larger than that of the 65% and 77% samples after annealing, indicating that the recrystallization behavior of the low strain samples did not occur at 700 °C. Even at different annealing times, the 65% deformation samples still contained larger grains, revealing that the deformation structure was still not uniform under low or medium strain. While the deformation increased to 77%, the number of coarse grains in the annealed samples decreased, and the fine grain and uniformity of grain size was further improved. For the same deformation in the steel wire, the average grain size decreased, or remained unchanged, with the increase in the annealing time.

Grains with a grain-orientation spread (GOS) less than or equal to 1° were generally considered recrystallized [23]. Three distinct stages were identified for the reversion of strain-induced martensite to austenite, which were followed by the recrystallization of the retained austenite phase and overall grain growth [10]. The austenite consisted of refined grains, though with different sizes and various colors, as highlighted in the figures, coarse elongated grains were present with the shape of the original cold drawing grains, as can be seen in figure 4. This inhomogeneity is due to a mixture of reversion-refined fine grains formed from deformation-induced martensite and recrystallized grains formed from deformed austenite. Furthermore, comparing with 0.4 and 1.0 cold drawing, sample of drawing strain 1.5 has more dislocation martensite structure, which is conducive to the nucleation of nanocrystalline/ultrafine austenite grains during annealing. On the other hand, the...
untransformed austenite in the drawing strain 1.5 sample has higher dislocation density and grain boundary density, which is also conducive to the formation of new defect free micron austenite grains by recovery recrystallization [24]. With the extension of annealing time, the new small austenite grains formed by martensite reverse transformation and original austenite recrystallization gradually grow (figure 4(i)).

The microstructures of cold-drawn stainless steel wires annealed at 800 °C for different times are shown in figure 5. For the three different annealing time samples annealed at 800 °C, the LAGB (low angle grain boundary) density was lower than that of the samples annealed at 700 °C, while the HAGB (high angle grain boundary) was higher than those at 700 °C, which indicates that recrystallization occurs more adequately at the high annealing temperature. As can be seen from figure 5, the grain size increased with the increase in annealing time at 800 °C for the same deformation wire. The grain sizes of the 33% deformation wires after annealing were obviously larger than those of the 65% and 77% deformations, which may be attributed to the fact that the driving force for the recrystallization and grain growth of the drawing strain 0.4 are lower than those in the 1.0 and 1.5. Compared with the 700 °C annealing temperature, the microstructure of the 800 °C sample had more uniformity, which could improve the ductility of cold-drawn stainless steel wire.

The localized deformation within the material can be observed from the kernel average misorientation (KAM). Figure 6 exhibits the KAM of the annealed wires with different deformation and annealing conditions. Green regions, which represent high misorientation, are distributed in the blue regions, which represent low misorientation. In the 33% deformation samples, there were still a lot of high misorientation regions after annealing at 700 °C with 20 and 60 min (figures 6(a) and (b)), which indicates that the heterogeneous structure of fine grains existed in the austenite matrix, and a large amount of plastic strain still existed in grain boundaries. However, with the increase in the strain of cold drawing, annealing temperature and time, the region of high...
misorientation in the annealing steel wires decreased. This shows that increasing the strain can promote the reverse transformation of deformed martensite and the recovery and recrystallization of untransformed austenite. But at 800 °C, even if the strain is less than 0.4, the reverse transformation of deformed martensite and the recrystallization of untransformed austenite occur by prolonging the time, and finally the grain grows and leads to grain coarsening. The reduction of high misorientation suggests that the structure were almost completely recrystallized during annealing. A small number of grains still had a high misorientation in the austenite matrix. Since the new austenite grains formed in the annealed sample with cold drawing strain 1.5 are more than those formed in the annealed sample with strain 0.4, this will inhibit the formation of coarse austenite grains (figures 5(a), (e) and 6(c), (k)). Therefore, the austenite structure of cold drawn strain 1.5 sample is more uniform after annealing. With the extension of annealing time, the fine austenite grains with high grain boundary density and high interface energy have thermodynamic instability, and then grow through grain boundary migration to reduce the grain boundary density. After long-time annealing, the austenite structure becomes coarse and uniform (figures 5(i) and 6(l)).

Those regions mainly correspond to the martensite and incomplete recrystallized grains in the cold-drawn austenite steel wire after annealing. For the 33% deformed wires, recrystallized action did not occur at 700 °C, but took place at 800 °C. However, sufficient recrystallization of stainless steel wires can occur when annealed either at 700 °C or 800 °C under large-strain drawing conditions, this is because high strain samples have a greater driving force for recrystallization. Thus, a DSC experiment was carried out to research the recrystallization starting temperature of cold-drawn stainless steel wires. The DSC curves of three steel wires with different deformations are shown in figure 7.

There are three peaks in DSC curves of the three deformed materials: the first peak can be attributed to the Curie temperature of δ ferrite [25]; the second peak can be attributed to the martensite reversion (α' → γ); and the exothermic peak may correspond to the recrystallization of the cold-drawn steel wire. The recrystallization starts temperatures of three steel wires with different deformation (33%, 65% and 77%) were 729 °C, 516 °C and 512 °C, respectively. Therefore, the steel wire with the 33% deformation was not prone to recrystallization when annealing at 700 °C (figures 4(a)–(c)). However, recrystallization should occur in the steel wires with different deformations when annealed at 800 °C.
3.3. Mechanical properties of samples before and after annealing

It can be seen from figure 8 that the strength of the cold-drawn stainless steel wires decreased with the increase in the annealing temperature and time (figure 8(a)). The trend of elongation of the cold-drawn steel wire was opposite to that of its strength (figure 8(b)). With the increase in annealing time and temperature, the volume fraction of the martensite decreased (figure 8(c)), while the fraction of the twin increased markedly (figure 8(d)). This high-density twinning may be attributed to annealing twins formed during the annealing process. The volume fraction of the recrystallized grain and the average grain size increase, with the increase in annealing time and temperature (figures 8(e) and (f)) such as at 800 °C, is higher than that at 700 °C. Meanwhile, the recrystallized grain volume fraction increased with the increasing of the strain or annealing time. Except for the cold drawing of the 33% deformation at 700 °C, when the deformation increased from 65% to 77%, the recrystallized grain volume fraction showed less sensitivity to the annealing time. From the above analysis, in contrast to the annealing time and temperature, the average size of recrystallization grains in the stainless steel wires has a stronger correlation with the deformation degree, which is related to storage energy.

4. Discussion

4.1. Microstructure evolution during annealing

The deformation microstructures of austenitic stainless steel wires cold drawn at room temperature usually include dislocation, stacking fault, twin and deformation-induced martensite, as shown in figure 3 and Refs. [20, 26]. The microstructure of the annealed stainless steel wires is given in figure 9.

As shown in figure 9, the annealed microstructures of the austenitic stainless steel wires still had plenty of dislocations, stacking faults, twin and deformation-induced martensite, but their morphology and quantity were different from that of the deformed microstructure. It is well-known that the microstructural characteristics are a major factor determining the mechanical properties of steels. The deformation-induced martensite structures are reversed into austenite structures, and the recrystallized austenite grains nucleate and grow into the deformed austenite structures during annealing [8], which is consistent with the research results in this paper (figures 8(c) and (e)). However, the reverse transformation temperature of martensite back to austenite during annealing is related to the thermal stability of martensite. Singh [27] introduced the thermal stability of martensite obtained after cold deformation of AISI 304 austenitic stainless steel. The results show that, when the annealing holding time after cold rolling is 1 h, the martensite can remain stable below 400 °C. Guy et al [28] studied the thermal stability of α′-martensite formed during cooling of 18%Cr-8%Ni steel and the results show that the α′-martensite is almost completely reversed back to austenite after holding at 600 °C for 2 min. In this study, the 304 austenitic stainless steel wire with a drawing strain of 1.5 was annealed at 800 °C for 60 min, and the martensite could not be completely reversed to austenite. Martensite reverse transformation temperature depending on the various criteria such as ultrafine-grained size, carbon content, and strain rate [6,7]. In addition, the reversal transformation from martensite to austenite usually has two mechanisms, namely the shear mechanism and diffusion control mechanism [28–30]. Those reversion mechanisms also mainly depends on the annealing temperature, the heating rate, and the chemical composition of the material. A lower Ni/Cr ratio leads to increase in the austenitization temperature for the martensitic shear reversion, while at very low Ni/Cr ratio, the martensitic shear reversion can no longer occur [29]. Under the
action of the shear mechanism, the reversed austenite has the same high density dislocations as the deformation-induced martensite, and the high-density dislocations rearrange to form sub-grains, which eventually leads to grain refinement. Under the diffusion dominated mechanism, the austenite nucleus nucleates and grows at the deformation induced martensite structures. A high Ni/Cr ratio and large deformation can promote the occurrence of shear mechanisms [29]. In this study, the Ni/Cr ratio of 304 austenitic stainless steel was about 0.44. Zhang et al [31] found that during the annealing process of austenitic stainless steel, some austenite structures that are reversely transformed with the shear mechanism will retransform into martensite during cooling. Meanwhile, the stacking fault energy (SFE) is closely related to the formation of martensite in austenitic steel wire during cold drawing [32]. A lower SFE is favorable when forming the nucleation site of martensitic transformation. A higher SFE decreases the mobility of extended dislocations and, thus, inhibits α’ martensitic transformation. The SFE of 304 austenitic stainless steel can be estimated according to the following equation [33]:

$$\gamma_{SFE}^{RT} = -53 + 6.2\text{Ni} + 0.7\text{Cr} + 3.2\text{Mn} + 9.3\text{Mo}$$  \hspace{1cm} (1)

$$\gamma_{SFE} = \gamma_{SFE}^{RT} + 0.05(T-20)$$  \hspace{1cm} (2)

where RT refers to room temperature, and T is in °C. SFE of austenitic steels is a function of alloy composition and temperature. It has been determined from experiments and semi-empirical simulations [34–36] However,
SFE is difficult to measure precisely, many researchers [37] measured the SFE for 301LN type steels by x-ray diffraction and obtained the average value of about 14 mJ m$^{-2}$. This value is close to that calculated from the composition based equation of Brofman and Ansell [34]:

$$\gamma_{\text{SFE}} = 16.7 + 2.1\text{Ni} - 0.9\text{Cr} + 26\text{C}$$

These regression equations indicate certain relationships between SFE and alloy composition. However, Vitos et al [38] calculated the SFE of austenitic stainless steels using a quantum mechanical first-principles approach and they demonstrated that the same alloying element can cause totally opposite changes in the SFE of Cr-Ni alloys with different host composition. This means that no universal composition-based equations for the SFE can be established. It can also be mentioned that the effect of nitrogen, an important alloying element, on the SFE of austenitic stainless steels is still a controversial issue, as discussed by Gavriljuk et al [39].

Figure 9. The TEM microstructure of annealed steel wires with different annealing conditions. $\varepsilon = 0.4$ cold-drawing samples annealed at 700 °C for 60 min (a)–(c), and at 800 °C for 20 min (d)–(f) and 40 min (g); and $\varepsilon = 1.0$ cold drawing samples annealed at 700 °C for 40 min (h) and at 800 °C for 40 min (i), respectively.

In general, the lower the SFE, the wilder the dissociation of partial dislocation and, thus, a stacking fault (SF) could be observed (as shown in figure 9(d)). In addition, SFs can also act as a precursor to the martensite, revealing that SFs can transform into martensite more easily when the deformation increases. The higher the SFE, the more unstable the martensite is. According to the results of the SFEs at 700 °C and 800 °C annealing, the martensite at 700 °C was more stable than that at 800 °C. Therefore, some martensite may reverse into austenite during the 800 °C annealing (figure 8(c)). It is worth noting that the volume fraction of the martensite increased with the prolongation of the annealing time at 700 °C for the annealing samples (figure 8(c)). Some studies [40] have explained this abnormal phenomenon. One explanation is that the recovery mechanism causes the stress elimination around the martensitic lath during annealing, so that the growth of martensitic lath can occur during cooling. The other explanation is that carbide precipitates are generated at the
gran boundary, which increases the temperature of MS, so martensite will be formed during cooling, resulting in an increase in the volume fraction of total martensite. These two phenomena may both occur in this study, which reasonably explains the abnormal increase of martensite with time annealing at 700 °C. In addition, the fraction of recrystallization under 700 °C and 65% deformation decreased with an increasing annealing time, which is contrary to the fraction of martensite under the same annealing conditions (figures 8(c) and (e)). The martensite was observed after annealing at 700 °C for 60 min for the 33% cold-drawn samples by TEM (figures 9(e), (f)). Compared with the collapsed martensite in the larger deformation samples at 800 °C, the straight shape of martensite and twinning was observed within the 33% deformation samples after annealing at 700 °C for 60 min (figure 9(c)). At high temperatures (800 °C) or high strain of 1.5, the recrystallization behavior was dominant, and the volume fraction of martensite decreased due to the reversion of martensite and recrystallization of deformed austenite [10]. The fraction of the martensite in the strain 1.0 annealed steel wire was the highest compared to other deformation and annealed samples. The inhomogeneous distribution of the recrystallized grains led to the formation of coarse grains (figure 4). The larger the deformation is, the lower the recrystallization temperature is. Therefore, in order to obtain a uniform and suitable grain size, a reasonable deformation and annealing temperature and time are required. In figure 8(e), it can be seen that an increase in annealing temperature significantly reduced the critical nucleation size of recrystallization, and the low strain of the cold-drawn steel wire still had a high recrystallized grain volume fraction at the higher temperature (800 °C). At 800 °C, the average grain size of low strain (0.4) annealed steel wire was higher than that of the high strain annealed steel wire, which was due to the high dislocation density and martensite in the high strain steel wire providing more nucleation sites, thus promoting grain refinement, while the recrystallized grains in the low strain steel wire engulfed adjacent grains and grew. (figure 8(a)).

4.2. Effect of microstructure on mechanical properties of annealed steel wire
During annealing, grain size and the volume fraction of the martensite have important influences on mechanical properties such as strength and elongation [41, 42]. The transformation induced plasticity (TRIP) effect in cold-drawn steel wire enhances the ductility of a material and necking during delayed strain, and the increase of the martensite volume fraction significantly enhances strength and reduces elongation [43]. Some results of tensile tests at different strains under different annealing regimes are shown in figures 8(a) and (b). It is obvious that various combinations of tensile strength, and elongation are possible to achieve. The tensile strength of the cold drawing 304 steel was very high, reaching the level of 979 MPa to 1551 MPa from strain 0.4 to 1.5, but the elongation was only 1% to 12%. However, in the regression process, the tensile strength and elongation of drawn steel wires with different strains show different trends. The results show that the tensile strength and elongation of annealed samples are 716 MPa to 826 MPa and 5.2% to 42% respectively. Obviously, with the extension of annealing time, the strength of annealed samples decreases and the elongation increases.

The average grain size of the drawn steel wire increased, while the tensile strength decreased significantly with the increase in annealing time at 700 °C of strain 0.4 (figure 8(a)). This is mainly due to the decrease in the small-angle grain boundary (2°–5°) density in the cold-drawn wire, with the increase in annealing time. Thus, dislocations and small-angle grain boundaries in the austenitic stainless steel can significantly improve the tensile strength of the material [44, 45]. When the strain increases from 0.4 to 1.0, the tensile strength increases. At this time, the grain refinement caused by martensite reverse transformation improves the tensile strength. The high tensile strength of strain 1.0 sample is attributed to grain size effect governed by Hall-Petch relationship. The lattice dislocation motion is blocked by high density of grain boundaries in the fine crystal sample. [46]. Furthermore, when annealed at 800 °C, a small-sized grain (figure 8(f)) and high martensite volume fraction (figure 8(c)) led to the increase in tensile strength when the strain increased from 0.4 to 1.0. It is well known that grain boundary has a profound effect on the tensile strength of steels leading to the well-known Hall-Petch relationship. Meanwhile, tiny martensite laths played a role similar to that of grain boundaries and effectively enhanced the tensile strength in the fine crystal sample [41].

However, when the strain increased from 1.0 to 1.5, the volume fraction of the martensite decreased in annealing at both 700 °C and 800 °C (figure 8(c)), while the average size of the grain did not change significantly (figure 8(f)), which is inconsistent with the increasing trend of the tensile strength (figure 8(a)). Recent studies have shown that a uniform grain-size distribution can improve the strength and ductility of austenitic stainless steel [8]. Figure 10 shows the grain-size distribution of the steel wires annealed at 800 °C for 60 min with strain rates of 1.0 and 1.5, where it can be seen that the grain-size distribution of the high strain (1.5) steel wire was more uniform after annealing at the same temperature and time.

Meanwhile, the annealing twins increase (figure 8(d)) with the strain increases from 1.0 to 1.5. Some researchers [47] point out that the boundary of annealing twin, similar to the grain boundary, is also considered as a barrier to dislocation motion. Thus, the high density of boundary of grain and twin did effectively restrict the movement of dislocations and caused high tensile strength in strain 1.5 sample.
In addition, at 700 °C, deformation degree is the key factor that influence the ductility, owing to the elongation increase from 9.8% at strain 0.4 to 39% at strain 1.0 after annealing for 60 min. Numerous low and high angle grain boundaries formed in the metastable austenite and some γ-phase transformed into strain-induced martensite during tensile straining which contributed to excellent elongation (TRIP effect) [41].

When the strain 0.4 specimen is annealed at 800 °C, the annealing time becomes the main factor affecting the elongation. When the annealing time lasts from 20 min to 60 min, the elongation increases from 13% to 40% (figure 8(b)), because the increase of annealing time promotes the further inverse transformation of martensite, which will reduce the ductility as a hard second phase. However, with the annealed duration increases at 800 °C, the tensile strength and elongation of a strain 1.5 specimen first decreases and then increases with the annealed duration increases, and further researches are needed for verifying whether this phenomenon is related to the density of movable dislocations [48].

In brief, as shown in figure 8, compared the tensile strength and elongation of specimen with three deformation degree, intermediated annealed sample is beneficial to further cold drawing for its relatively good match between strength and elongation. After annealing at 700 °C for 60 min of strain 1.0 specimen, the elongation reached 39%, while the tensile strength is just 776 MPa. When annealing at 800 °C for 20 min with strain 1.5, the tensile strength reached 810 MPa and the elongation reached 40% (figures 8(a) and (b)). Based on the cost consideration of industrial production, specimen with strain 1.5 annealed at 800 °C for 20 min is the suitable regime for the further cold drawing processing, because a feasible heat treatment process in a relatively short time is obtained balancing the tensile strength and elongation.

5. Conclusions

By means of DSC, EBSD, TEM and mechanical testing, the microstructure and mechanism properties were analyzed in annealed cold-drawn 304 austenitic stainless steel with different rates of strain (0.4, 1.0 and 1.5), annealing times (20, 40 and 60 min) and temperatures (700 °C and 800 °C). The effects of the deformation and annealing process parameters on the microstructure and mechanical properties were described comprehensively in this paper. The main conclusions of this paper are as follows:

1. The tensile strength of commercial 304 austenitic stainless steel wire is increased from 659 MPa to 1551 MPa, through the drawing deformation of strain from 0–1.5, and the elongation is reduced from 45% to 1%, showing excellent cold working ability.

2. The differences in the deformed structures, annealing temperatures and times led to the different kinetics of recrystallization and martensite transformation during the annealing process, resulting in different microstructure evolution and mechanical properties. The recrystallization temperatures are determined by the degree of strain, while the stability of martensite in steel wires is determined by the stacking fault energy.
and drawing parameters. With the increase in strain, the temperature of recrystallization and martensite reversal decrease accordingly.

3. The content of martensite and twin in the stainless steel wires are related to annealing temperature. Compared to 700 °C, the volume fraction of the martensite decreased and the annealing twins increased in the steel wire at 800 °C, which is beneficial to the improvement of ductility in steel wire.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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