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Effect of cryogenic rolling and annealing on the microstructure evolution and mechanical properties of 304 stainless steel

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Abstract: Metastable 304 austenitic stainless steel was subjected to rolling at cryogenic and room temperatures, followed by annealing at different temperatures from 500 to 950°C . Phase transition during annealing was studied using X-ray diffractometry. Transmission electron microscopy and electron backscattered diffraction were used to characterize the martensite transformation and the distribution of austenite grain size after annealing. The recrystallization mechanism during cryogenic rolling was a reversal of martensite into austenite and austenite growth. Cryogenic rolling followed by annealing refined grains to 4.7 μm compared with 8.7 μm achieved under room-temperature rolling, as shown by the electron backscattered diffraction images. Tensile tests showed significantly improved mechanical properties after cryogenic rolling as the yield strength was enhanced by 47% compared with room-temperature rolling.

Keywords: stainless steel; cryogenic rolling; annealing; microstructural evolution; mechanical properties; recrystallization

1. Introduction

Austenitic stainless steels (SS) usually exhibit excellent corrosion resistance and good formability. However, they have a relatively low yield strength of about 200 MPa in the annealed state because of the presence of a soft face-centered cubic γ-austenite phase [1–2]. Therefore, these materials are less suitable for structural applications. Various strengthening mechanisms have been applied, such as grain refining, transformation strengthening, and work hardening [3]. Among these, grain refining simultaneously improved the strength and toughness of austenitic stainless steels [4]. Severe plastic deformation (SPD) is a popular method for obtaining a fine grain structure but introduces large structural defects into bulk specimens [5]. The common SPD processes used for bulk materials include equal-channel angular pressing [6], high-pressure torsion [7], and accumulative roll bonding [8].

Grain refinement in austenitic stainless steels may be accelerated during SPD because of the formation of deformation twins in metastable γ-austenite-grains and subsequent stress-induced phase transition [9–13]. Some research has shown that the volume fraction of martensite is strongly dependent on pre-strain, strain, strain rate, stress state, and temperature [14–16], and also on the stacking fault energy (SFE) of SS [17].

Microstructure evolution and its effect on the mechanical properties of austenitic stainless steel treated with SPD and subsequent annealing have been studied by several researchers [18–20]. Shen *et al.* [18] reported that an ultrafine-grained 304 SS was prepared by using accumulative rolling and annealing at 550°C for 150 s. The recrystallized γ-grains were 270 nm in size. The ultra-high yield strength and ultimate tensile stress reached 1870 and 1950 MPa, respectively. However, the loss of the elongation to failure is serious at only 6% ± 1%. Kumar and Raabe [19] performed cyclic annealing of a 90% cold-rolled 304L SS at 900°C for 45 s and 850°C for 45 s, and produced γ-grains in size of 300–2000 nm with a yield strength of 710 MPa and a tensile ductility of 36%. Similarly, a high yield strength of about 1000 MPa with a high plasticity (about 32%) has been achieved in a 93% cold-rolled and annealed 304 SS [20].

Recently, cryogenic rolling (CR) has been used to prepare nanocrystalline alloys with a low stacking fault energy

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(SFE) [21–23]. Severe rolling at subzero temperatures helps accumulate a high density of defects and restricts dynamic recovery, which in turn promotes a high density of nucleation sites during controlled annealing and results in a finer grain structure [21–24]. Therefore, deformation twinning and stress-induced martensitic transformation are expected to occur in 304 SS with CR, which may promote extensive grain refinement. Roy *et al.* [25] reported the synthesis of nanostructured austenitic AISI 304L SS after CR and reversion annealing at 700–800°C, and discussed the effect of processing parameters on the evolution of bulk nanostructure and its influence on tensile properties. However, they did not discuss the changes in properties and structure. Although Li *et al.* [26] and Xiong *et al.* [27] studied the structure and mechanical properties of 310 SS after CR and annealing, and found that stress-induced martensitic transformation did not occur during deformation because the stable γ-grain in 310S did not undergo stress-induced phase transition. A few of researches exist on the recrystallization process. Our previous study discussed the change in microstructure and its influence on properties during cryogenic deformation [28]. In this work, the evolution of structure and its influence on properties during recrystallization were discussed with the aim of enriching the theories and application of CR.

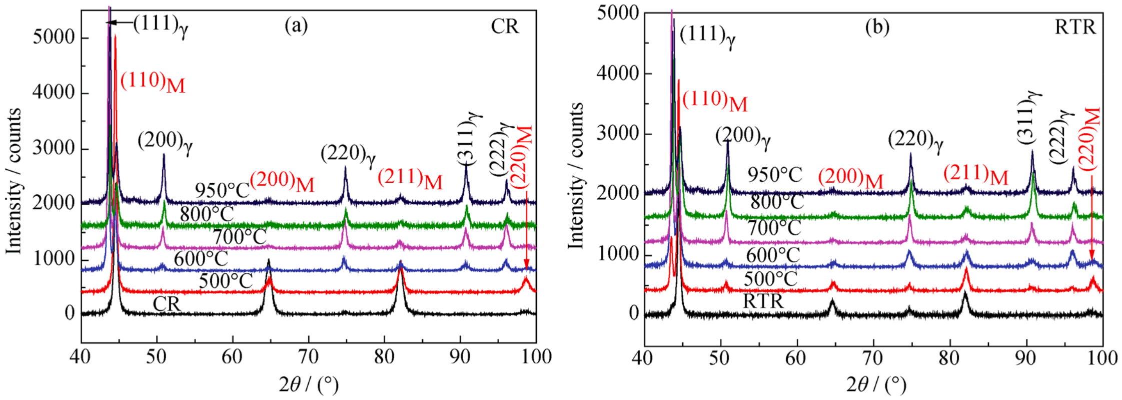
2. Experimental

Long bars (100 mm × 30 mm × 9.5 mm) of 304 austenitic stainless steel (17.75Cr–1.12Mn–8.2Ni, wt%) were solution-treated at (1100 ± 5)°C for 1 h then water-quenched. Cryogenic deformation (or rolling) (about 10%–90% thickness reduction) was performed by immersing the plates in liquid nitrogen for at least 20 min before each rolling pass. The as-quenched samples were rolled at room temperature and were termed room-temperature rolled (RTR) samples. The samples with a reduction of 50% were annealed at about 500–1050°C for 5 min followed by room-temperature cooling. The bulk hardness of all specimens was measured at ten different locations using a Vickers hardness tester (401 MVD) with a load of 200 g for a residence time of 15 s and a Rockwell hardness tester. Structural characterization was performed using an Axio Imager A2 optical microscope. Samples were prepared according to GB/T 4334.1—2000. Microstructural details were studied using a JEMS-2010 transmission electron microscope (TEM) at 200 kV. TEM samples were ground mechanically to 40–60 μm thick and then thinned using a twin-jet thinning method with an electrolyte of 10vol% perchloric acid and 90vol% ethanol at −20°C and about 50–75 mV. Metallurgical cross-sections were cut from the gauge of the deformed samples and were evaluated for martensite volume fraction using X-ray diffractometry (XRD, SmartLab) analysis. The detailed calculation method is available in Refs. [29–32]. A scanning electron microscope (SEM, ZEISS SUPRA 55) with electron backscattered diffraction (EBSD) was used to collect micrometer scale crystallographic information on the annealed specimens. Data acquired by EBSD were evaluated using HKL software Channel 5. Tension tests were conducted at room temperature according to the American Society for Testing and Materials (ASTM) E8/E8M-11 Standard using a Reager 3010 tensile machine with a loading rate of 1 mm/min. The mechanical properties were averaged from three individual tests.

3. Results and discussion

3.1. XRD analysis

The deformation microstructure of CR and RTR 304 stainless steels with different reductions has been described in detail [28]. The volume fraction of martensite from CR steel with a reduction of 50% became 92vol%, whereas that of the RTR was 81vol%. Fig. 1 shows the XRD patterns of the 304 stainless steels with 50% reduction under various conditions. Figs. 1(a) and 1(b) show the XRD spectra of CR and RTR 304 stainless steel with 50% reduction after annealing at different temperatures. No change in phase structure can be observed at 500°C. An increase in annealing temperature leads to a gradual transformation of martensite into austenite. At 600°C, this situation changes significantly and γ-austenite phase peaks appear. When the temperature reaches 700°C, the α′-martensite phase peak obviously decreases. The calculated results indicate that the martensite fraction decreases with increasing annealing temperature. For example, the volume fraction of martensite in the CR sample was 92vol%, however, it decreased to 18vol% after annealing at 950°C. In addition, the volume fraction of martensite did not decrease, but increased in the CR sample at 500°C. The sample was full martensite containing $(110)_M$, $(200)_M$, $(211)_M$, and $(110)_M$ peaks without any trace of γ. It is believed that annealing causes a stress release, which triggers shear in the retained austenite and leads to a transformation to martensite [33]. In the CR and RTR samples, the general trend in martensitic transformation was to initially decrease rapidly then decrease slowly, as depicted in Fig. 1(c).



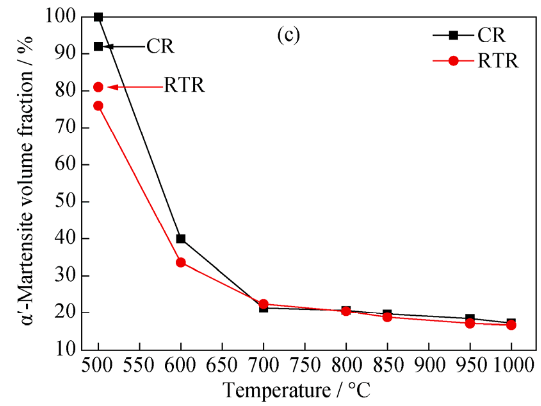


Fig. 1. XRD patterns of CR (a) and RTR (b) 304 stainless steels with 50% reduction after annealing at different temperatures (γ—austenite, M—martensite); volume fraction of martensite (c) of CR and RTR 304 stainless steels with 50% reduction after annealing at different temperatures.

3.2. TEM investigation

Fig. 2 shows the TEM micrographs of the 50% rolled samples annealing at 500–700°C for 5 min. With an increase in annealing temperature, the typical rolling microstructure transforms gradually into a recrystallization structure through the recovery and recrystallization process. Finally, recrystallized equiaxed austenite grains with clear grain boundaries are formed. However, the different rolled microstructures in CR and RTR steels lead to a different recovery and recrystallization process. Fig. 2(a) shows the TEM image of the CR steel after annealing at 500°C. The microstructure is still the martensite lath with an obvious boundary, as shown in Fig. 2(a). The martensite in the CR steel did not recover to the austenite phase until the annealing temperature increased to 600°C. At this point, although the martensite partly retained its lath geometry, it began to transform into small austenite grains of about 110 nm or less. The high density of dislocations in the martensite itself caused dislocation motion, resulting in the semi-martensite rearranging to form sub-grains, as shown in Fig. 2(b). When the annealing temperature of the CR steel increased to 700°C, the dislocation density inside the grains began to decrease visibly, whereas the equiaxed recrystallized grains began to appear in large numbers, as shown in Fig. 2(c). Statistical analysis of the equiaxed grains in the TEM diagram indicates that the recrystallized grain size ranged from 100 to 700 nm, as shown in Fig. 2(c). Some retained fine lath martensite is shown in Fig. 2(c). Because the CR samples with 50% reduction were almost all martensite, the annealing process resulted in the reverse transformation of martensite and the subsequent recovery and recrystallization process.

However, the RTR samples with a reduction of 50% contained martensite and a considerable portion of austenite. This recrystallization was more complex than CR after annealing, especially above 600°C. Annealing treatment at 500°C showed little change in microstructure, as shown in Fig. 2(d). The two different sources of austenite at 600°C were the original austenite and reversed austenite, as shown in Fig. 2(e). Because of the characteristics of the high density dislocations in the martensite, the reversed austenite also contained high density dislocations. This led to the recovery and recrystallization of reversed austenite because recrystallization is driven by a high density of dislocations. At this time, however, this process did not occur in the original austenite as the low-density dislocations could not provide sufficient driving force. The phenomenon of lath martensite and austenite having different degrees of dislocation density has been reported in detail [28,34]. For the RTR samples, an increase in annealing temperature to 700°C yielded equiaxed grain recrystallization, with low dislocation density, at the site of the original martensite but not in the original austenite regions. The austenite maintained high density dislocations, as shown in Fig. 2(f). It is concluded that the recrystallization process only occurs at a higher annealing temperature compared to 700°C. However, grains that had completed recrystallization grew at a higher annealing temperature.

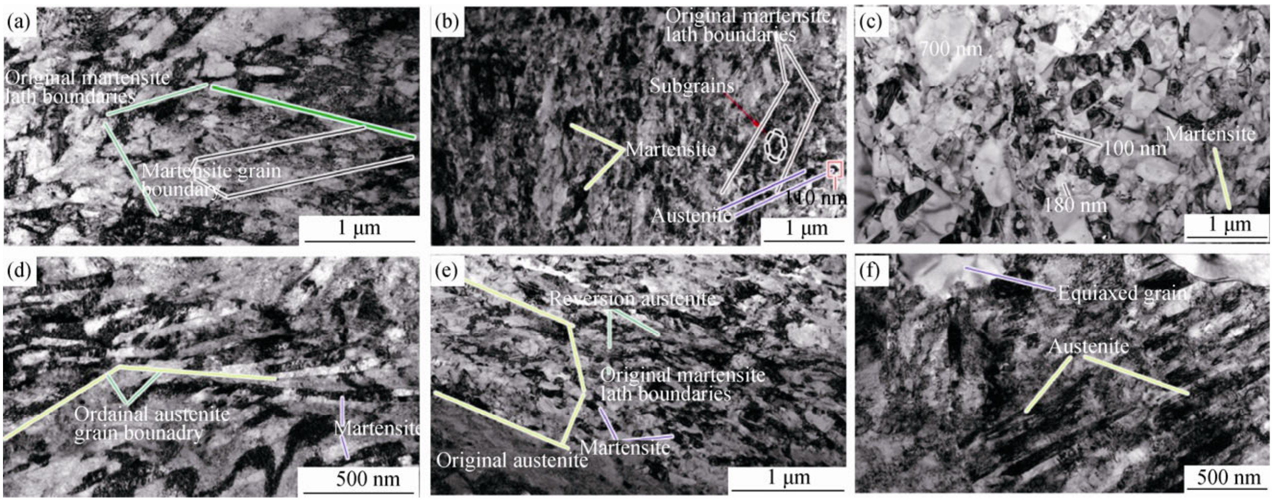


Fig. 2. TEM images of CR (a, b, c) and RTR (d, e, f) 304 stainless steel after annealing at 500°C (a, d), 600°C (b, e), and 700°C (c, f).

3.3. EBSD characterization

A series of EBSD measurements were used to measure the variation in grain size for the rolled samples annealed for 5 min at 800, 850, 900, and 950°C. Figs. 3 and 4 show the grain morphology and misorientation after annealing at 800–950°C. In the CR samples, although all grains exhibited almost complete recrystallization at 800°C, a large number of low-angle grain boundaries (LAGBs) exist. Figs. 3(a)–3(c) show that the grain size did not change with an increase in annealing temperature. The grain size distribution was more uniform than the RTR sample. The increase in annealing temperature caused a change in grain misorientation, as shown in Fig. 4(a), where the LAGBs decreased and the high-angle grain boundaries (HAGBs) increased. However, in the RTR samples, the misorientation and grain size have no change after annealing at 800°C. It can be inferred from these results that the recrystallization temperature of RTR steel must be higher than that of the CR steel. This

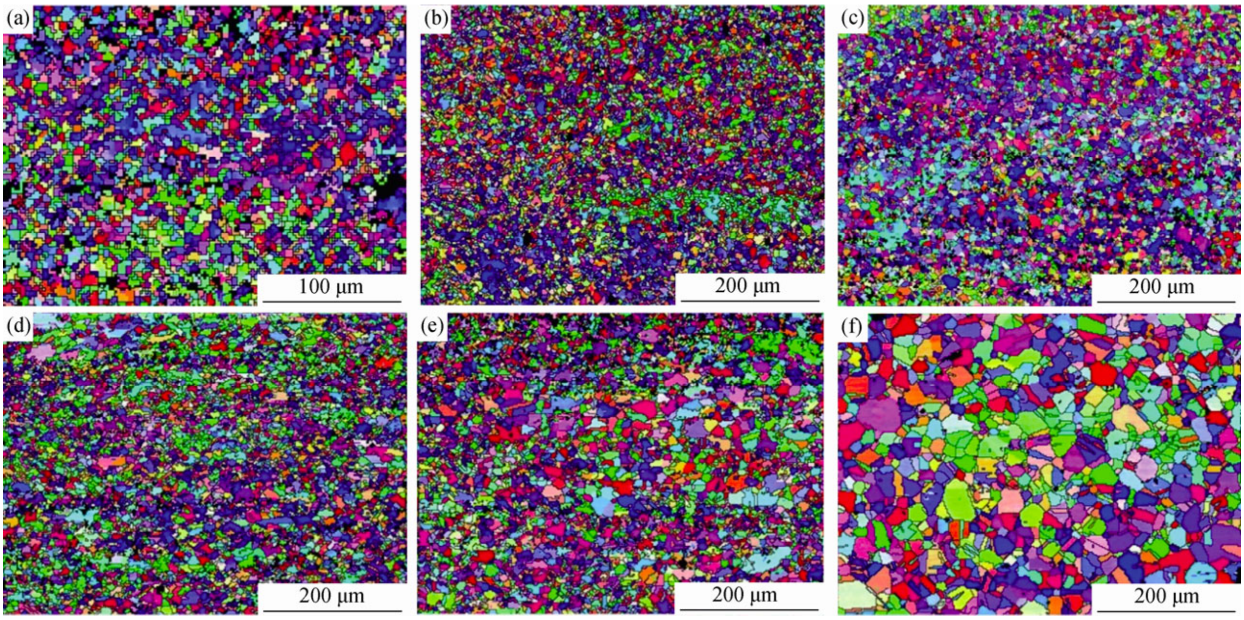


Fig. 3. EBSD images of CR (a, b, c) and RTR (d, e, f) 304 stainless steel after annealing at 800°C (a), 850°C (b, d), 900°C (c, e), and 950°C (f).

indicates that after annealing at the same temperature, the RTR samples exhibited a greater residual deformation stress than the CR samples. Because the annealed RTR samples at 800°C were difficult to collect data by EBSD, the EBSD measurements were used to measure the variation in grain size for the three samples annealed at 850, 900, and 950°C. Fig. 3(d) shows a weak bimodal structure. This phenomenon is more obvious when the annealing temperature increases to 900°C, as shown in Fig. 3(e). The LAGBs decrease and the HAGBs simultaneously sharply increase, as shown in Fig. 4(b). When the annealing temperature continues to increase, the misorientation no longer changes. However, the recrystallization grain size increases significantly and the average grain size develops from about 4.7 to 8.7 μm, as shown in Figs. 4(c)–4(d). The main reason is that the grain size of the gap was too large. During annealing, large grains gradually engulf surrounding finer grains and fine grains merge to form large grains. Therefore, as the number of small grains decreases the number of large ones increases [35].

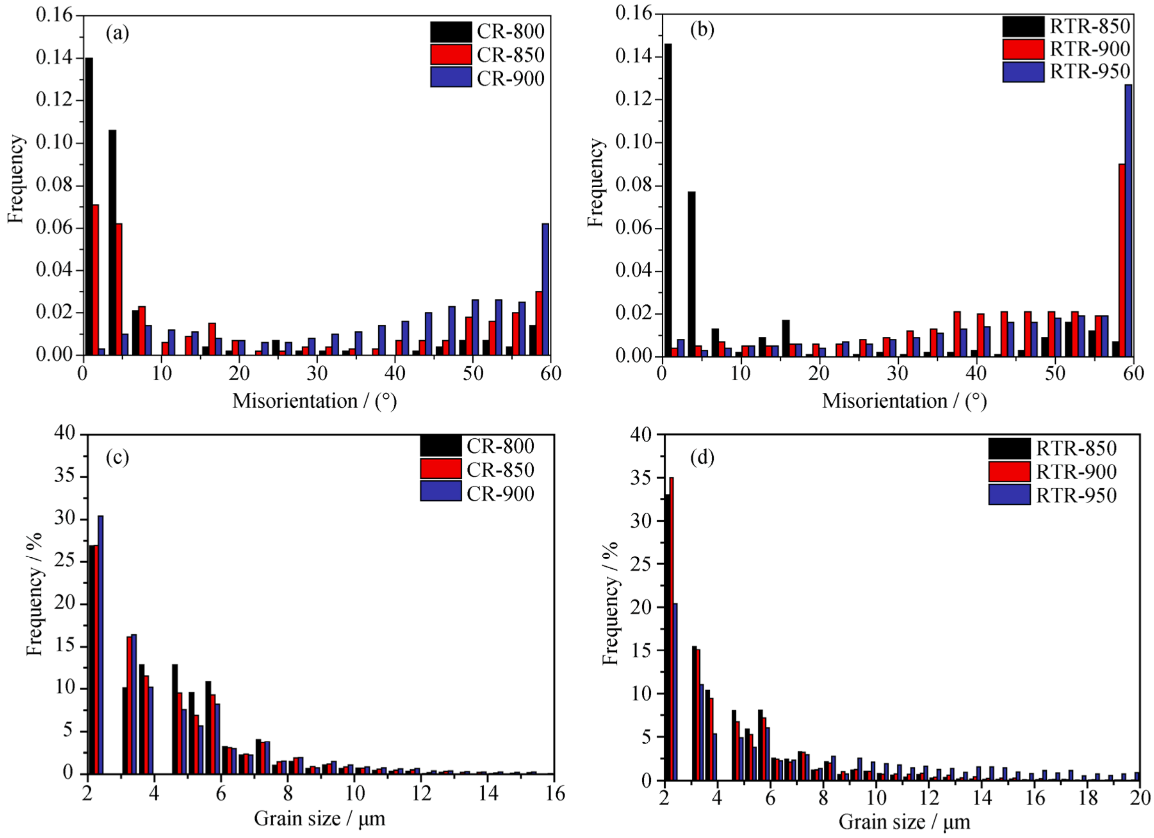


Fig. 4. Grain misorientation plots of CR (a) and RTR (b) 304 stainless steels after annealing; grain sizes of CR (c) and RTR (d) 304 stainless steels after annealing (CR-800: CR with annealing at 800°C).

3.4. Tensile deformation

Figs. 5 and 6 show the engineering stress–strain curves, the mechanical properties, and the Vickers hardness of the CR and RTR samples after annealing at different temperatures. The rolled samples showed high strength and low elongation-to-failure values, as evident in Fig. 5. The CR and RTR samples exhibited similar property values, because there was no obvious change in microstructure after annealing at 500°C. With an increase in annealing temperature, the strength and hardness decreased and the elongation increased. Two mutation points existed at 600 and 700°C during the process. At 500°C, the CR sample exhibited a yield strength (YS) of 1711 MPa, a tensile strength (TS) of 1708 MPa, and an elongation (EL) of 4.4%. However, the YS and TS of the CR sample decreased, respectively, from 1711 to 1343 MPa and from 1708 to 1236 MPa, whereas the EL increased from 4.4% to 7.4%. RTR-600 (RTR with annealing at 600°C) exhibited a similar change in mechanical properties. It appears that the main reason for the change in mechanical properties in CR-600 and RTR-600 is martensite reverting to austenite and its grain recrystallization. As stated previously, the martensite reversal and recrystallization process continues at 700°C. This leads to a further decrease in strength and significant improvement in elongation; the EL of CR-700 increased approximately four times (from 7.4% to

30.4%). With an increase in grain size from 100–800 nm to 1–4 μm, the change in mechanical properties is consistent with the above description, regardless of the process. Some differences exist between the two deformation processes. For example, in the CR sample, when the annealing temperature exceeded 800°C the grain size remained stable (at about 4 μm), therefore the TS remained at about 820 MPa and the YS at about 360 MPa. However, the grain size in RTR-950 doubled compared with that in RTR-900, from about 4 to 8 μm, which led to a reduction in YS to about 248 MPa. It can be seen from Fig. 6(b) that the hardness did not change after annealing the CR sample above 900°C, as the grain size and tensile properties stabilized at this temperature. Therefore, a recrystallization temperature of about 900°C is confirmed for CR steel. The same method was used to determine the recrystallization temperature of RTR steel at 950°C.

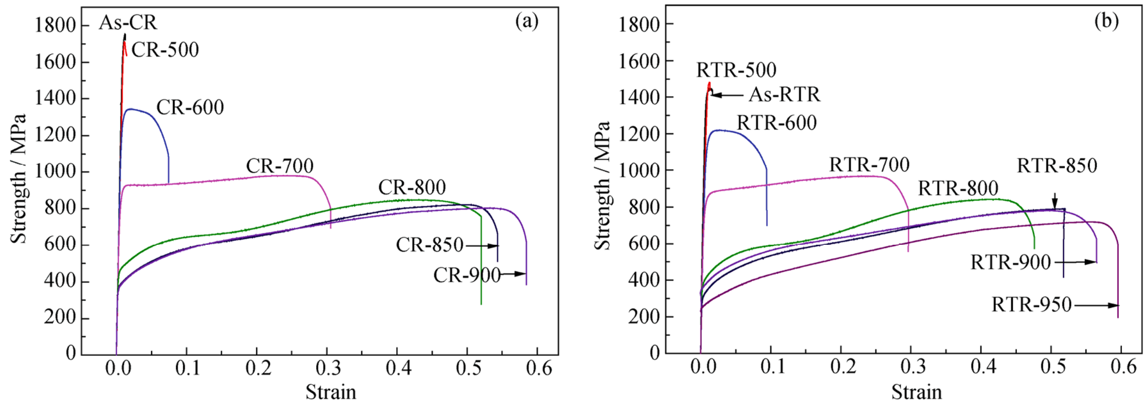


Fig. 5. Engineering stress–strain curves of CR (a) and RTR (b) 304 stainless steels after annealing at different temperatures.

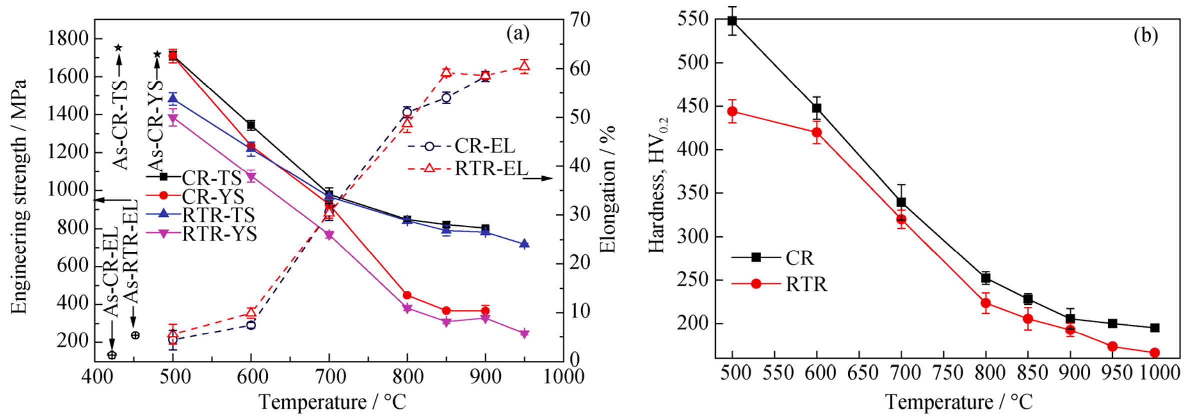


Fig. 6. Mechanical properties (a) and Vickers hardness (b) of CR and RTR 304 stainless steels after annealing at different temperatures.

4. Discussion

The deformation mechanism of austenitic stainless steel is influenced strongly by its SFE [10,17]. An accumulation of different types of defects, i.e., dislocations, twins, and stacking faults, and their densities in an evolved sub-structure are influenced strongly by the severity of rolling and temperature. Different deformation types produce different microstructures [28]. CR steel contains predominantly martensite and RTR steel is a mixture of martensite and austenite, as shown in Fig. 1. Different structures may lead to different recrystallization mechanisms, which results in different grain sizes. Finally, refining the grain structure improves the mechanical properties of the metal.

In this study, XRD analyses revealed that an α′ → γ transformation occurred in the samples annealed at 600°C for 5 min. Forouzan *et al.* [36] reported that a complete conversion of α′ → γ occurred in cold-rolled 304L SS during annealing at 800°C for 4 min. Our TEM study confirmed that nearly equiaxed γ-sub-grains, with a mixture of HAGBs and LAGBs, were achieved in all samples annealed at 700°C. The resulting microstructure was inhomogeneous and comprised 100–700-nm ultrafine grains. Some literatures [37–38] explained that upon reversion annealing the γ-subgrain size depends on the thickness of the α′-laths, as the nucleation and growth of γ occur in strain-induced α′.

However, the phenomenon of inhomogeneous distribution is significantly improved in the subsequent annealing process, as shown in Figs. 3(a)–3(c). In RTR steel the difference in dislocation density between martensite and austenite is very large [34] and results in two types of phases that cannot synchronize recovery and recrystallization. For instance, one is already an equiaxed grain of about 500 nm and the other is a deformation organization with a high density of dislocations, as shown in Fig. 2(f). The low fractions of retained γ-austenite in the RTR samples act as nuclei, which grow rapidly during annealing in competition with fresh γ-nuclei in the α′-laths. This results in a bimodal grain structure, with the largest grain being over 10 μm and the smallest below 1 μm.

5. Conclusions

In summary, the effects of martensitic transformation after reversion annealing on the resultant austenite grain structure of a CR and RTR 304 stainless steel were studied. Important findings are as follows:

(1) A method of refining the grain size of 304 austenitic stainless has been known using a combination of CR and annealing. Compared with the recrystallization grain size of 8.7 μm achieved in the RTR steel, that of CR decreased to 4.7 μm. CR also significantly decreased the recrystallization temperature from RTR steel's 950 to 900°C.

(2) CR and RTR steels exhibit different recrystallization mechanisms. Because the structure is almost fully martensitic after 50% CR, the recrystallization mechanism of CR steel involves only the reversal of martensite into austenite and austenite growth. RTR steel follows the same process, along with the recovery recrystallization of residual austenite as a result of the coexistence of two types of phases. For CR steel, grain refinement is mainly dependent on the thickness of the α′-laths.

(3) Grain refinement significantly improves the mechanical properties of steel. The TS and YS of the recrystallized CR steel increased by 11% and 47% compared with those of the RTR steel (TS: 803.1 and 718.7 MPa; YS: 366.6 and 248.8 MPa, respectively), whereas the EL was not significantly reduced (CR: 58.4%; RTR: 60.0%).

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