The Effects of Martensite Thermomechanical Parameters on the Formation of Nano/Ultrafine Grained Structure in 201LN Stainless Steel

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Abstract

In this study, the effects cold rolling and annealing parameters during thermomechanical processing of the AISI 201LN stainless steel were investigated. The cast samples were homogenized, hot-rolled and solution-annealed to acquire a suitable microstructure for the subsequent thermomechanical treatment. Unidirectional and transverse multi-pass cold rolling at 25, 0 and -15 °C was carried out to 90% reduction in thickness, followed by annealing at temperature range of 700–900 °C for different times of 15–1800 s. Microstructures were characterized by optical and scanning electron microscopy, X-ray diffraction and feritscope measurements. Hardness test was also used for evaluating mechanical properties of the specimens. The results showed that the saturation strain of martensite formation during the cold rolling at 25, 0 and -15 °C in 201LN stainless steel was about 0.29, 0.23 And 0.17, respectively. A nano/ultrafine grained structure was formed by annealing the 90% cold rolled specimen at 800 °C for 60 s with a grain size of about 300 nm.

Keywords: Martensite thermomechanical processing, 201LN stainless steel, Strain-induced martensite, Nitrogen.

1. Introduction

In recent years, owning to the price and allergic reactions of nickel, a lot of works have been done to develop a new class of austenitic stainless steels with a low Ni content. In these steels, the Ni is balanced with Mn and N addition for keeping the austenitic microstructure. Nitrogen-alloyed austenitic stainless steels are new materials that have favorable mechanical properties such as high strength and ductility, desirable toughness and work hardening, unmagnetizability, good corrosion resistance and reduced tendency to grain boundary sensitization. All of these features are of great importance for sea water systems, automobile and nuclear industries, ballistic applications and many applications at ambient and cryogenic temperatures [1-3]. It is well confirmed that such a high efficiency results from the role of N in solid solution. Nitrogen is known as an austenite stabilizer, solid solution strengthener and it improves pitting corrosion resistance, fracture toughness, creep and fatigue strength [4].

When austenitic stainless steels are cold rolled, austenitic shear bands are formed owing to the

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enhanced planar slip. By increasing the cold rolling reduction, intersection between shear bands is occurred so that these shear band intersections are transformed to nucleation sites for martensite embryos [5]. More shear band intersections are generated with increasing cold rolling, resulting in nucleation and growth of the strain-induced martensite (SIM). Because of the shear bands to be consumed, only martensite exists in the specimens after heavy cold rolling [6]. It should be noted that the austenite stability, i.e., the tendency to shift to the SIM transformation, is dependent on chemical composition, temperature, strain state and the initial grain size of the austenite phase and strain rate [7].

The martensite thermomechanical treatment that is based on the SIM transformation is nowadays one of the main methods used for producing nano/ultrafine-grained austenitic stainless steels. This method involves cold rolling followed by reversion annealing of the SIM to austenite.

In most cases, the formation of the nano/ultrafine grain structure in the cold-rolled and annealed stainless steel like AISI 201 may cause $Cr_{23}C_6$ precipitation, making this steel susceptible to sensitization and intergranular corrosion. In this respect, the sensitization problem would be prevented in the AISI 201LN steel which has austenite stabilizer like nitrogen while it is deficient in carbon [6].

Although several studies have been conducted on the thermomechanical processing of austenitic stainless steels alloyed with nitrogen [1,6,7,8-10], no systematic studies have been conducted on the grain refinement of 200 series austenitic stainless steel

alloyed with nitrogen. The purpose of the present study is to investigate the effects of processing parameters of the thermomechanical treatment on the development of nano/ultrafine grain structure in the AISI 201LN austenitic stainless steel.

2. Materials and experimental procedures

The chemical composition of AISI 201LN stainless steel is shown in Table 1. This steel was prepared by induction melting in an atmosphere of N_2 gas. At first, the cast ingots were homogenized at 1200 °C for 4 h followed by water quenching. Then the ingots were hot rolled at 1100 °C. It has been reported that nitrides, such as Cr_2N, can completely dissolve into the austenite at 1075 °C [11]; hence solution annealing was carried out at 1150 °C for 2.5 h followed by water quenching. Unidirectional and transverse multi-pass cold rolling was carried out at room temperature, 0 °C and -15 °C with the thickness reduction of 0-90%. The annealing treatments were carried out at 800-900 °C for 15-1800 s. Figure 1 shows the thermomechanical processes used in this work to obtain the nanocrystalline structure. The phase composition and the amount of SIM during cold rolling and annealing were measured by X-ray diffraction (XRD, Philips X' Pert with Cu Kα anode) and Feritscope. The scan speed of XRD was 3 deg.min^{-1} and the voltage and current were 40 kV and 200 mA, respectively. The surface of specimens was electropolished at 25 V for 60 s by using an electrolyte containing 200 ml perchloric acid and 800 ml ethanol. Quantification of SIM was carried out by magnetic technique using portable Feritscope MP 30E-S, Fisher,

Germany, calibrated with standard test blocks before measurements. To verify the repeatability of the results, leastways of 8 measurements were made for each specimen. The Feritscope readings were multiplied by the correction coefficient of 1.71. Hardness of the samples was measured by Vickers method (HV) with the indenting load of 10 kg and the average of 5 indentations. Microstructural investigations of the specimens were carried out using optical and scanning electron microscopy (SEM Philips X230) techniques after electro-etching in 65% nitric acid solution. Grain size was measured by the line intercept method in accordance with ASTM E112.

3. Results and discussion

Figure 2(a) shows optical microstructure of the as-cast specimen, which represents an austenitic matrix with about 16% volume fraction of delta ferrite. After homogenization at 1200 °C for 4 h, the amount of delta ferrite was decreased to 12.8%. The optical microstructure of the as-homogenized specimen is shown in Figure 2(b). As shown in Figure 2(c), grain size was clearly reduced after hot rolling at 1100 °C. Figure 2(d) shows the optical microstructure of the specimen annealed at 1100 °C for 150 min. The solution annealed material exhibited an austenitic microstructure with equiaxed grains characterized by an average size of about 33 μm. As can be seen, the majority of the microstructure consisted of austenite while there was a low amount (about 3.5%) of the retained delta ferrite along the grain boundaries.

Table 1. Chemical composition of used AISI 201LN (wt. %)

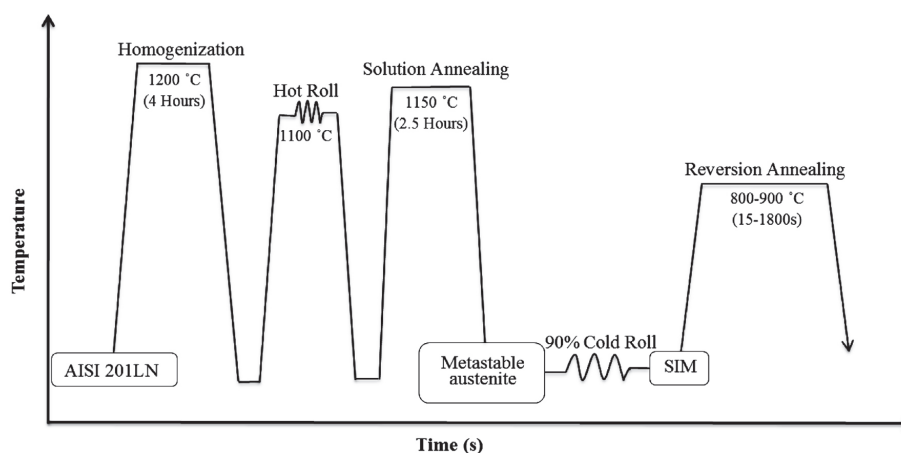| Alloy	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	N	Fe
AISI 201LN	0.03	0.34	5.90	0.033	0.011	16	0.109	3.91	0.15	0.1	Balance

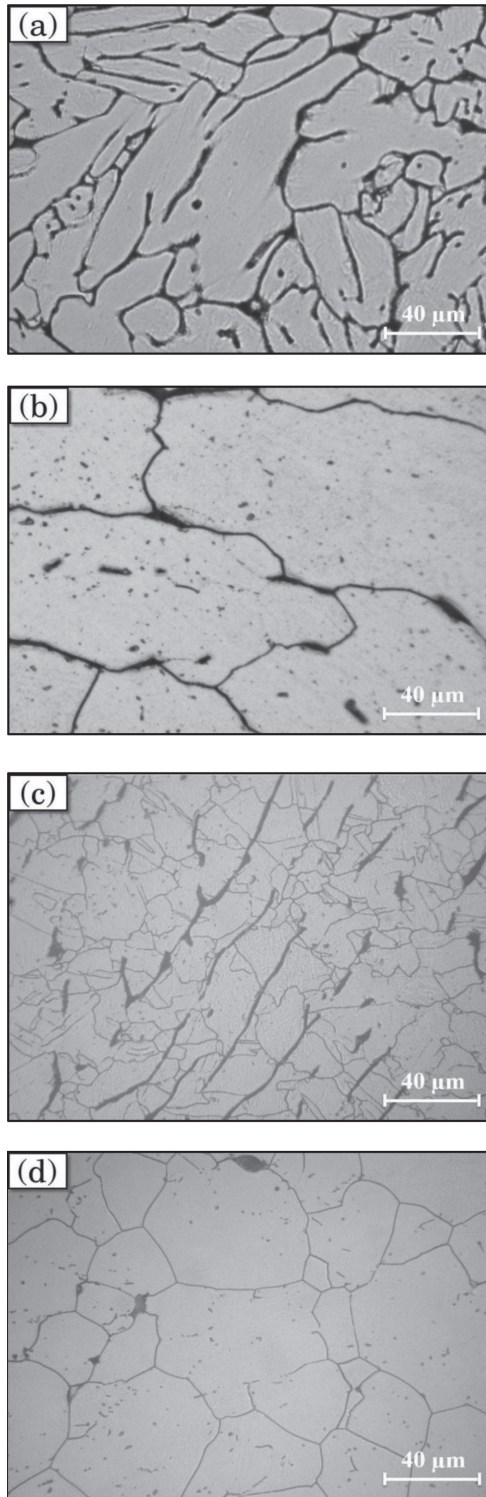*Fig. 1. The schematic diagram of the thermomechanical treatment used in this work.*

Fig. 2. Optical micrographs of the (a) as-cast, (b) as-homogenized, (c) as-hot rolled and (d) as-solution annealed specimens.

Figure 3 shows the cold rolled optical micrograph of the 201LN austenitic stainless steel specimens for different thickness reductions wherein ά-martensite phase are etched dark and the austenite grain boundaries are not etched. As shown in this figure,

with increasing the cold rolling reduction, ά-martensite was increased. Nucleation of ά-martensite phase occurred at intersections of shear bands and growth of ά-martensite resulted from the repeated nucleation of the new embryos. It should be noted that shear bands are planar defects produced from overlapping of stacking faults on austenite {111} planes during plastic deformation and the overlapping process determines the structure of the shear bands [7].
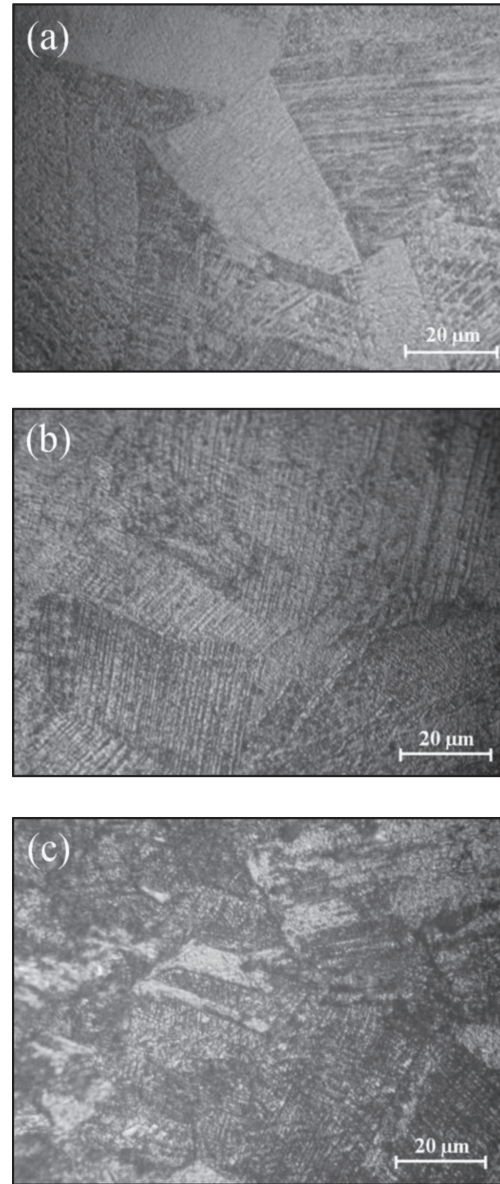

Fig. 3. Optical micrographs of the cold rolled specimens at 25 °C after thickness reductions of (a) 10%, (b) 20% and (c) 30%.

Figure 4 shows variations of the volume fraction of the SIM in the AISI 201LN stainless steel cold rolled at 25 °C as a function of thickness reduction. As can be seen, the volume fraction of the SIM was increased with increasing the thickness reduction and finally

microstructure became 100% martensite after about 25% thickness reduction (r), indicating that saturation strain ($\varepsilon_s = \ln(1/1-r)$) was 0.29. As shown, the AISI 201LN steel used in this work due to the existence of thermodynamically metastable austenite at room temperature was transformed to 100% martensite during cold rolling below the M_d temperature. This finding is in good agreement with previous works reporting that the 200 series austenitic stainless steel were completely transformed into the ά-martensite using cold rolling at room temperature [12-14].
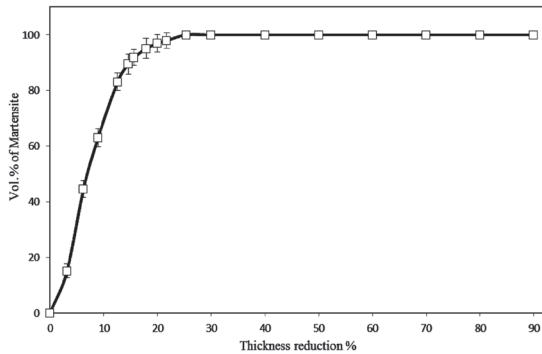

Fig. 4. The volume fraction of SIM as a function of thickness reduction.

The effect of thickness reduction on the variation of hardness in the AISI 201LN stainless steel cold rolled at 25 °C is shown in Figure 5. The hardness values were increased with cold rolling due to the formation of SIM and strain hardening resulting from increasing dislocation density. It should be noted that the rate of hardness variation before ε_s was high due to the formation of SIM; on the other hand, the rate of hardness increase was high after about 55% thickness reduction. This can be attributed to the martensite fragmentation [15].
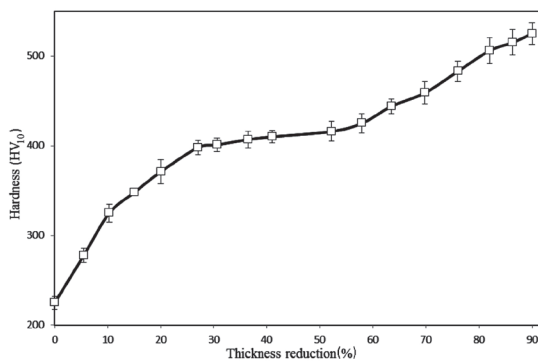

Fig. 5. The effect of thickness reduction on the hardness variation.

Figure 6 shows XRD patterns of the cold-rolled specimens with 20, 30 and 90% reduction at 25 °C. As can be seen, with increasing the cold rolling reduction, the intensity of austenite peaks was gradually decreased, and intensity of the martensite peaks was increased in the spectrums. Finally, the microstructure was changed to fully ά-martensite after 90% cold rolling.
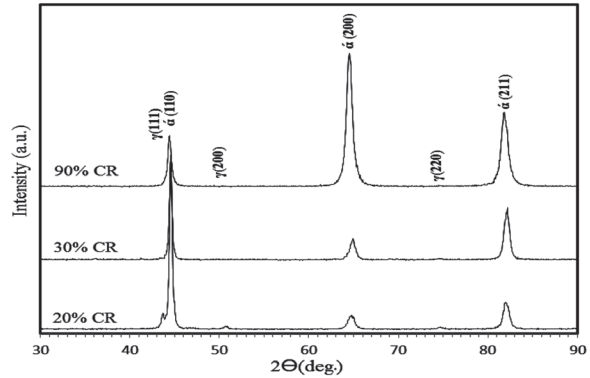

Fig. 6. X-ray diffraction patterns of the cold rolled specimens at 25 °C.

Figure 7 shows the effect of strain path on the formation of SIM. Due to an increase of the shear band intersection sites, the formation of martensite was easier at cross rolling as compared to conventional rolling and therefore, the amount of ε_s was smaller (e.g. 0.2 and 0.29 for the cross rolling and conventional rolling, respectively).
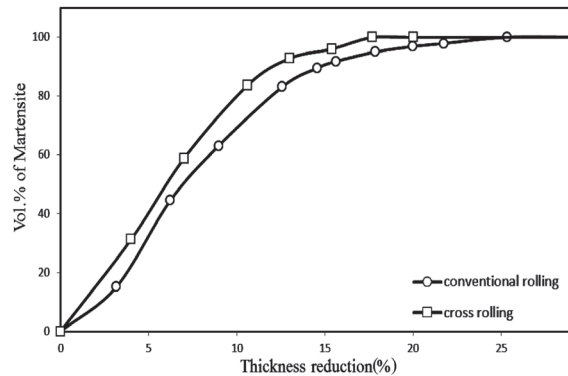

Fig. 7. The effect of strain path on the formation of SIM.

Figure 8 shows the volume percentage of the SIM during cold rolling at different temperatures. Cold rolled samples at 25, 0 and -15 °C had been transformed to 100% SIM after about 25, 21 and 16% thickness reduction, respectively and ε_s for them was about 0.29, 0.23 And 0.17, respectively. This means that with reducing cold rolling temperature, ε_s was reduced. The temperature dependence of the austenite stability and ά-martensite transformation arose from the temperature dependence of the stacking fault energy. The tendency for the shear band formation and the generation of the nucleation sites for the ά-martensite depend on SFE. Several authors have reported that with decreasing the temperature, the amount of SFE decreases [16-18] and therefore, driving force for SIM increases.
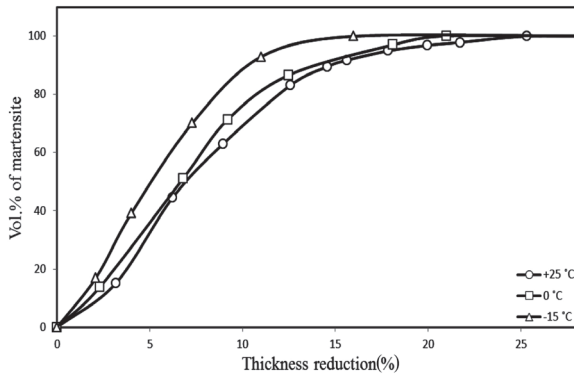
Fig. 8. The effect of rolling temperature on the formation of SIM.

The effect of the initial austenite grain size on the kinetic of SIM formation during cold rolling is shown in Figure 9. The ε_s value for the samples cold rolled with the average grain size of 21, 33 and 54 μm was 0.36, 0.29 and 0.24, respectively. As can be observed, increasing the initial austenite grain size decreased the ε_s and made the SIM transformation faster. These results are in agreement with the fact that with increasing austenite grain size, the critical stored energy for the formation of the SIM is decreased. This means that decreasing the grain size makes the austenite more stable and increases the M_{d30} temperature [19, 20].
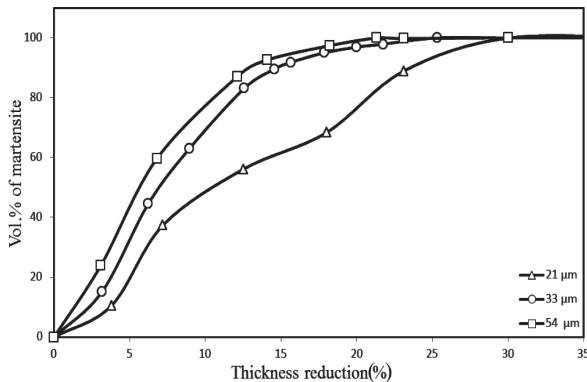

Fig. 9. The effect of initial austenite grain size on the formation of SIM.

Figure 10 shows the effects of annealing time on the amount of austenite reversion in the samples after 90% reduction in thickness at 25 °C, followed by annealing treatment at the temperature range of 800-900 °C for 15-1800 s. As can be seen, the reversion rate was increased

for the annealing in the higher temperatures. For example, the martensite reversion was not observed after 30 s annealing at 800 and 850 °C, while the measured martensite volume fraction was only about 7.5% at 900 °C. A complete reversion occurred at 800, 850 and 900 °C after about 90, 75 and 45 s, respectively. As shown in the figure, at low annealing temperature and short annealing durations, the austenite nucleation did not occur, while at higher annealing temperatures or longer annealing durations, it happened, suggesting a possible diffusion mechanism for the reversion of ά-martensite to austenite in this study [6]. It is also shown that a secondary increase in martensite can be seen at higher annealing times especially at 850 and 900 °C. The M_s temperature is a beneficial parameter for estimating the stability of austenite against martensitic transformation. The calculated M_s temperature of the test material by Eichelmann and Hull [21] was -26 °C. If it is imagined that due to the precipitation of carbides, total carbon content of the steel in the austenite matrix of the steel has been depleted, M_s temperature is increased to 24 °C. This means that the austenite stability is decreased by depletion of the matrix from carbon owning to an increase in M_s temperature. Thus, the formation of thermally-induced martensite (TIM) during quenching after annealing may seem logical. Increasing martensite volume fraction during annealing was reported by some authors [13, 22, 23].
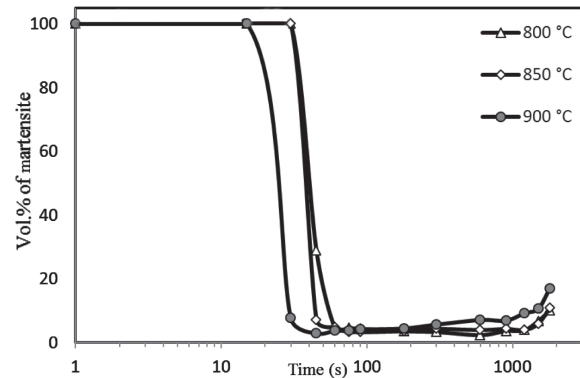

Fig. 10. The effects of annealing time on volume fraction of the martensite at different temperatures.

The effect of annealing time at 800, 850 and 900 °C on the hardness values is shown in Figure 11. As can be seen, hardness decreased with increasing temperature and annealing time. Some authors [14, 24] attributed this to the decreasing of dislocations density, volume fraction of martensite in the structure and grain growth.
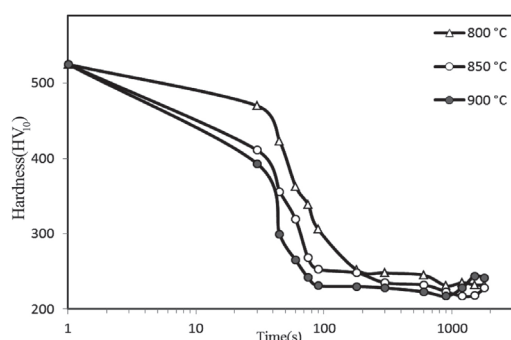
Fig. 11. The effects of annealing time on hardness values at different temperatures.

The microstructures of the specimens annealed at 800°C for 60 and 1800s after cold rolling at 25°C are shown in Figure 12. As can be seen, annealing at 800°C for 60 s resulted in the smallest grain size of about 300 nm with 95% austenite reversion (Figure 12(a)). Increasing the annealing time caused grain growth so that grain size after annealing at 800°C for 1800s reached to 3 µm (Figure 12(b)).
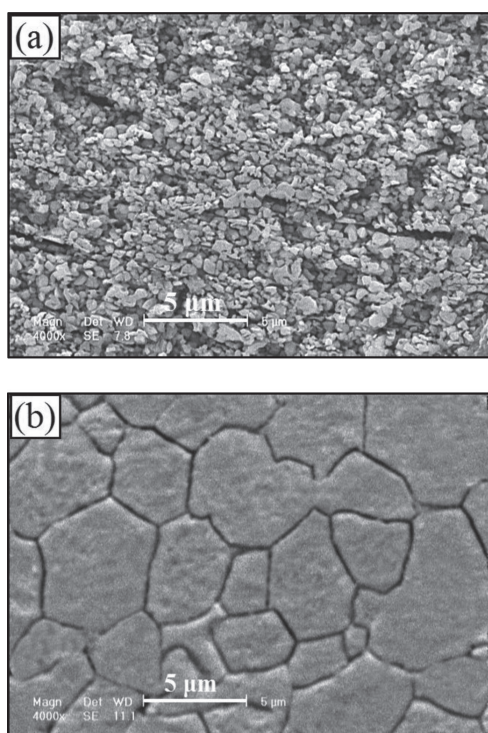

Fig. 12. The microstructure of the specimens annealed at 800 °C for (a) 60 s and (b) 1800 s after cold rolling at 25 °C.

4. Conclusions

The thermomechanical processing of a 201LN austenitic stainless steel subjected to severe cold rolling and subsequent annealing was studied. The main results can be summarized as follows:
1. The saturation strain of martensite formation during cold rolling at room temperature in 201LN stainless steel was about 0.29.

2. Increasing the initial austenite grain size and strain, and decreasing cold rolling and applying cross rolling instead of conventional rolling promoted the formation of SIM.
3. Formation of TIM after annealing was due to the carbide precipitation and the increase of M_s temperature.
4. Annealing at 800 °C for 60 s resulted in the smallest grain size of about 300 nm with 95% austenite reversion.

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