

The Effects of Nitrogen Addition on the Production of Ultrafine/Nano Grained AISI 201L Stainless Steel by Advanced Thermo-mechanical Process

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Abstract

In this study, the effects of nitrogen addition on the production of ultrafine/nano grained AISI 201L stainless steel by advanced thermo-mechanical process were investigated. Cast samples were first homogenized at 1200 °C for 4 h, hot-rolled at 1100 °C and finally, solution-annealed at 1150 °C for 2.5 h. Unidirectional and cross multi-pass cold rolling at 25 °C was carried out to 90% reduction in thickness, followed by annealing at temperature and time range of 800–900 °C and 15–1800 s, respectively. The results showed that increasing nitrogen led to the decrease of delta ferrite content and austenite grain size after solution annealing. It was also found that decreasing nitrogen content and using cross rolling resulted in an increased volume fraction of the strain induced martensite (SIM) and reduced saturation strain of martensite formation during cold rolling. Also, the results from the reversion process revealed that with increasing nitrogen concentration, the formation of thermally induced martensite was suppressed.

Keywords: Martensite thermomechanical processing, Nitrogen, 201L stainless steel, Cold rolling, Reversion annealing.

1. Introduction

Stainless steels are a main class of engineering materials used in a wide variety of industries and environments. Austenitic stainless steels are the most common and familiar types of stainless steels exhibiting attractive properties such as high formability, good corrosion resistance and low ductile-to-brittle transition temperatures. Austenitic stainless steels show the best corrosion resistance and ductility among the stainless steels. However, they have a relatively low strength, particularly yield strength, restricting their structural applications. Among the different strengthening mechanisms, grain refinement is the only method used to improve both strength and toughness at the same time¹⁻³.

Severe plastic deformation and advanced thermomechanical processing are the most important techniques to produce ultrafine grained steels. For the SPD techniques, a well-designed strain path is more important and also more feasible than a

precisely controlled temperature path. The small scale, complexity and the discontinuous nature of these processes propose that they would require considerable ingenuity and investment to be applied on a high-volume industrial scale. Compared with severe plastic deformation methods, the advanced thermomechanical processing routes are less effective in terms of grain refinement, but they are more efficient with respect to mass production. The advanced thermomechanical processes are continuous and they can be easily optimized⁴.

Austenitic stainless steels are unstable during deformation, transforming into α' -martensite^{5,6}. Several studies have been conducted on the fabrication of Ultrafine/Nano grain austenitic stainless steels alloying with nitrogen⁷⁻¹², but there is no systematic study on the effects of nitrogen addition on the grain refinement of 201L austenitic stainless. The present paper was intended to comprehensively understand the effects of nitrogen addition and processing parameters of the thermomechanical treatment on the development of nano/ultrafine grain structure in the AISI 201L austenitic stainless steel.

2. Materials and experimental procedures

Laboratory ingots of nitrogen alloyed 201L stainless steel containing 0.16 and 0.23%wt nitrogen were prepared by induction melting in an atmosphere of N₂ gas. The chemical compositions of the alloys studied are given in Table 1.

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Table 1. Chemical compositions of steels used in this investigation (wt. %)

Alloy	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	N	Fe
N16*	0.03	0.57	5.60	0.023	0.013	16.3	0.094	4.22	0.02	0.16	Balance
N23	0.03	0.56	5.61	0.034	0.013	16.2	0.094	4.31	0.02	0.23	Balance

*Note: The symbol N16 indicate this alloy has 0.16%wt nitrogen

The cast ingots were homogenized at 1200 °C for 4 h followed by water quenching; then they were hot rolled at 1100 °C. Solution annealing of the ingots 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 with the reduction of 90%. The deformed samples were subjected to inter-pass water cooling to avoid the effect of heating during rolling. The annealing treatment was carried out at 800-900 °C for 15-1800 s. The schematic thermomechanical processes used in this work have been shown in Figure 1.

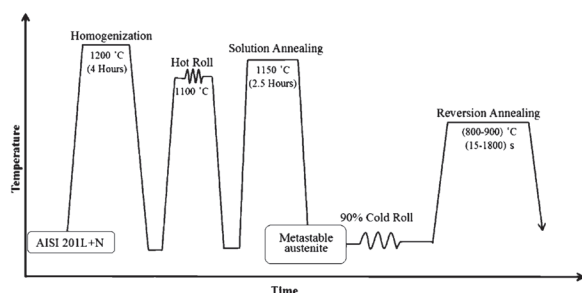


Fig. 1. The schematic diagram of the thermomechanical treatment used in this work.

The phase composition was measured by XRD using Philips machine with Cu K α anode. 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 electro-polished at 25 V for 60 s using an electrolyte containing 200 ml perchloric acid and 800 ml ethanol. Quantification of martensite in each sample was carried out by the magnetic technique using portable Feritscope MP 30E-S, Fisher, calibrated with α -ferrite standard samples 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 1.7. Hardness of the samples was measured by Vickers method (HV) with the indenting load of 10 kg. Microstructural investigations of the specimens were carried out using optical microscopy after electro-etching in 65% nitric acid solution.

3. Results and discussion

Figure 2 shows optical microstructures of the specimens annealed at 1100 °C for 150 min. The solution annealed steel with 0.16 %N, here called N16, exhibited an austenitic microstructure with a low

amount of the retained delta ferrite along the grain boundaries, whereas the optical microscopy of the as-solution annealed steel with 0.23 %N, here called N23, showed the fully austenitic microstructure. It is well known that nitrogen is a strong austenite stabilizer, thereby reducing the amount of delta ferrite. As can be seen in this figure, austenite grain size of N16 and N23 specimens were 32 and 27 μ m, respectively. The results showed that with increasing nitrogen content, the austenite grain size was decreased.

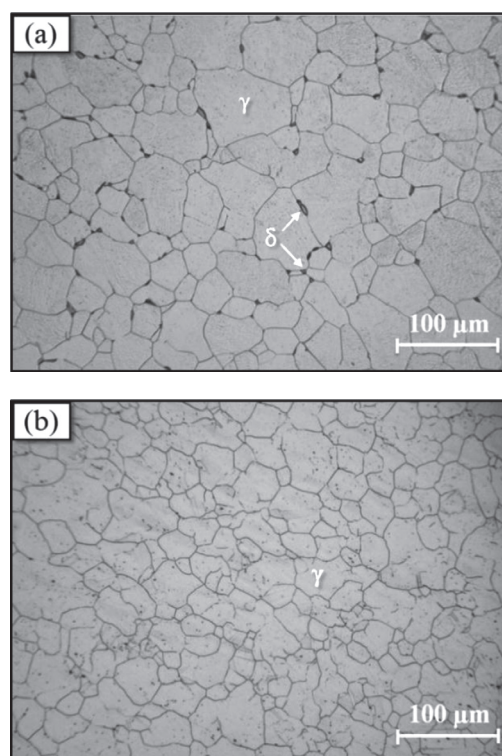


Fig. 2. Optical micrographs of the as-solution annealed specimens with nitrogen contents of: (a) 0.16% and (b) 0.23%.

After solution treatment, unidirectional and transverse multi-pass cold rolling was carried out with the thickness reduction of 90%. Figure 3(a) shows variations of the volume fraction of the martensite in the AISI 201L stainless steels N16 and N23 as a function of thickness reduction. As can be seen, the volume fraction of the SIM was increased with increasing the thickness reduction; finally, after 90% thickness reduction, microstructure in N16 and N23 samples was composed of 95 and 70% martensite phase, respectively. This means that with

increasing nitrogen concentration, austenite stability and stacking fault energies (SFE) were increased¹³ such that transformation to martensite during cold rolling was retarded. The SFE played an important role in determining the austenite stability because it controlled formation of the shear bands, hence the formation of nucleation sites for the α -martensite¹³.

The effect of thickness reduction on the variation of hardness in N16 and N23 steels has been shown in Figure 3(b). The hardness values were increased with cold rolling reduction, which was due to the formation of martensite and strain hardening resulting from increasing dislocation density. Hardness in N16 steel was increased from 237 to 581 HV as the cold rolling reduction was increased from 0 to 90%, whereas in N23 steel, it was increased from 252 to 642 HV. The effect of nitrogen on strength and hardness was due to the misfit between the interstitial nitrogen atoms and the octahedral lattice voids. This led to strains in the surrounding lattice, and also the increase of shear stress due to nitrogen interaction with dislocations¹⁴.

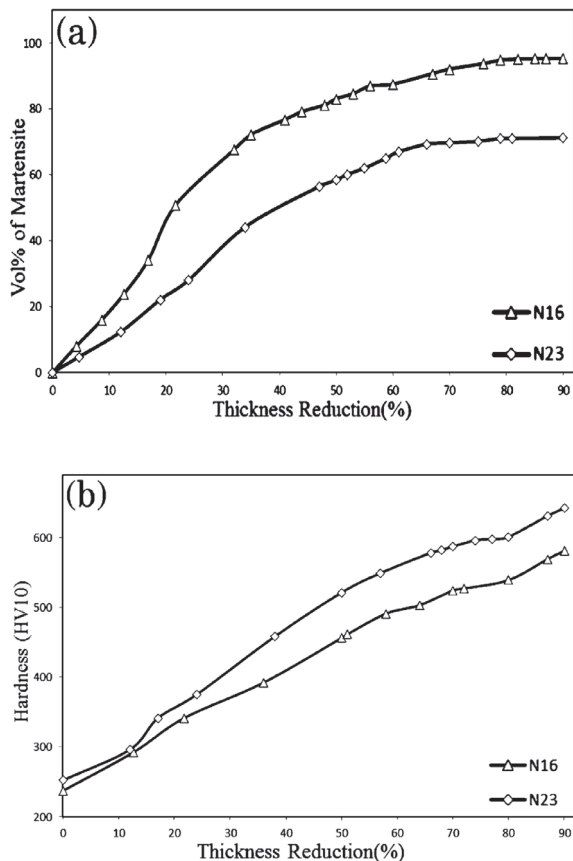


Fig. 3. The effect of thickness reduction on the: (a) volume fraction of the martensite and (b) hardness values.

Figure 4 shows the effect of strain path on the formation of martensite. It is observed that the cross rolled steels showed a greater content of α -martensite

at each strain than the conventionally rolled ones because of the increase in shear band intersection sites. For example, cross rolled N16 samples were transformed to 100% martensite after about 60% thickness reduction, whereas conventional rolled samples were converted to 95% martensite after 90% thickness reduction. In N23 steel, cross rolled and conventional rolled samples after 90% thickness reduction were transformed to 81 and 71% martensite, respectively.

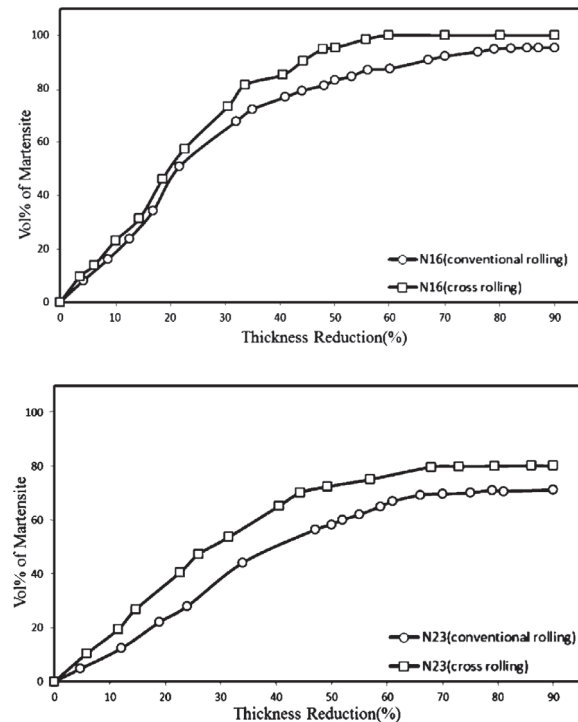


Fig. 4. The effect of thickness reduction and strain path on the volume fraction of the martensite.

Figure 5 shows the XRD patterns of the specimens after 90% cold rolling. The XRD pattern of the N16 specimen shows only martensite reflections, whereas N23's pattern shows martensite and austenite peaks.

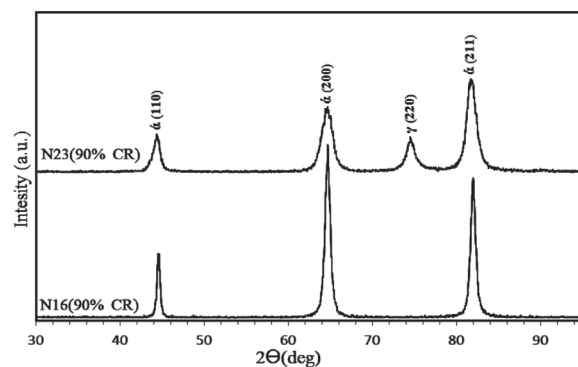


Fig. 5. XRD patterns of the specimens with nitrogen contents of 0.16% and 0.23% after 90% cold rolling.

The effect of annealing time on the amount of austenite reversion for different temperatures in the specimens with different nitrogen contents is shown in Figure 6. As can be seen, the reversion rate is faster for higher annealing temperatures, especially for short periods of annealing. Also, in this figure, the secondary increase in the martensite content as long periods of annealing is negligible, whereas the previous study¹⁵⁾ showed an increase in martensite content in an AISI 201L containing 0.08 and 0.1%wt nitrogen as annealing for 1800 s. This may be attributed to the effect of nitrogen on austenite stability and the decrease of M_s temperature and subsequently, the decrease of thermally induced martensite¹⁶⁾. After reversion annealing at 850 °C for 30 s, N16 and N23 samples contained 97 and 99% austenite, respectively.

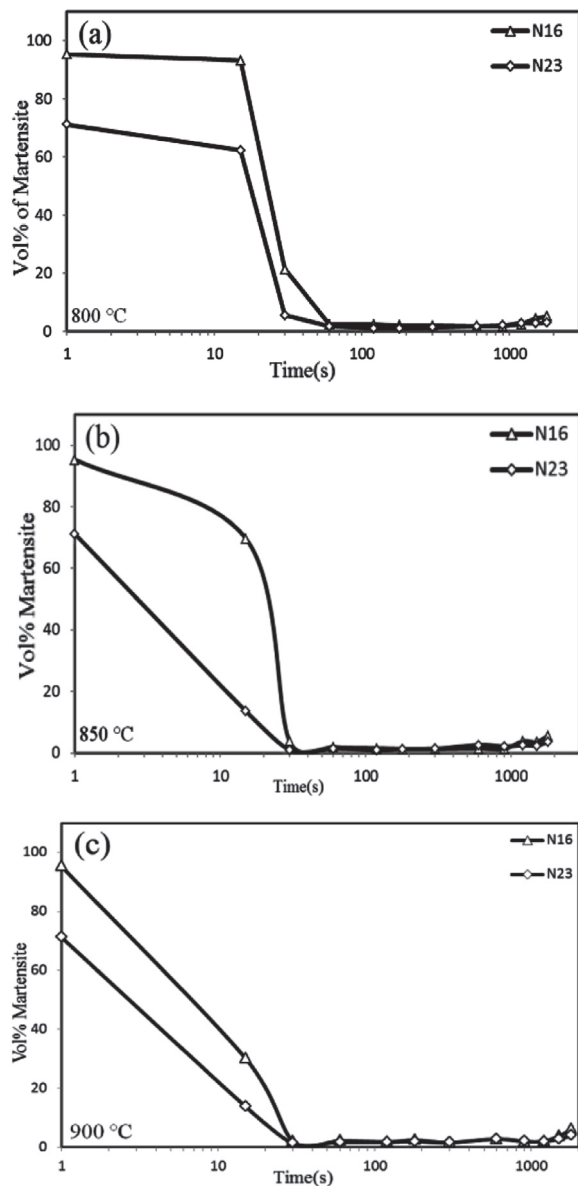


Fig. 6. The influence of annealing temperature on the martensite of N16 and N23 steels at: (a) 800, (b) 850, (c) 900 °C.

Figure 7 shows the dependence of hardness versus annealing time and temperatures in N16 and N23 steels. As can be seen, the hardness values decrease with increasing the annealing time and temperature. This is related to the lower density of dislocations, the lower volume fraction of martensite in the structure and the grain growth caused by increasing the annealing time and temperature. It should be noted that increasing the N content increased hardness values.

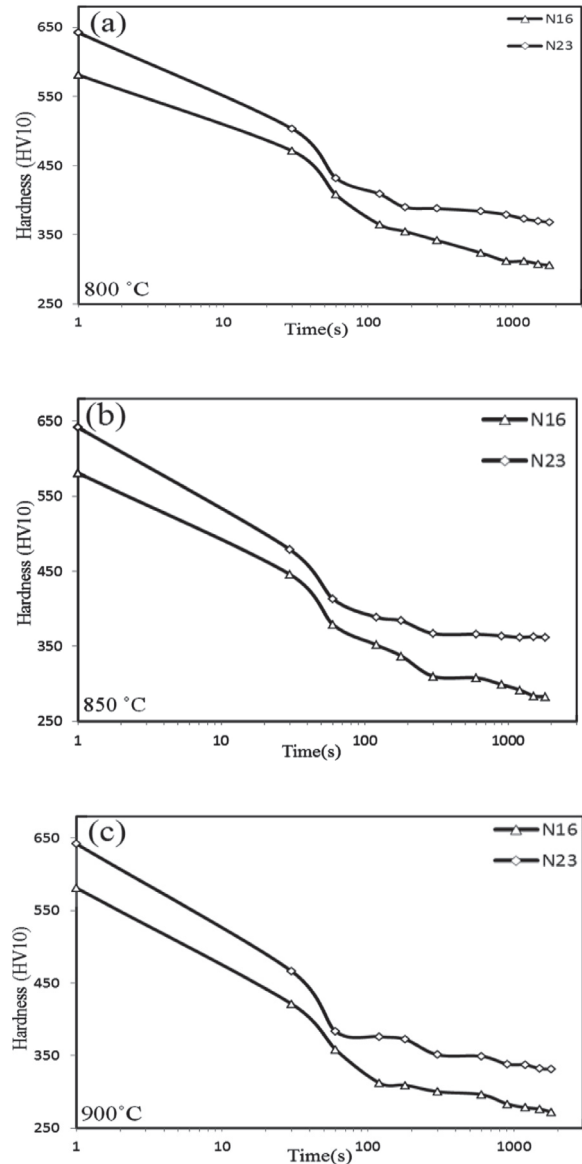


Fig. 7. The influence of annealing temperature on the vickers hardness values of N16 and N23 steels at: (a) 800, (b) 850, (c) 900 °C.

4. Conclusions

In the present investigation, the effect of nitrogen on mechanical and microstructural properties of AISI 201L stainless steel with 0.16 and 0.23 %wt N content during solution annealing, cold rolling and

reversion annealing was characterized. The following conclusions can be drawn from this work:

1. After solution annealing, the austenite grain size and the amount of delta ferrite were decreased with increasing the N content.
2. Decreasing the N content and applying cross rolling instead of conventional rolling promoted the formation of martensite.
3. The reversion rate was much faster at higher annealing temperatures.
4. Nitrogen avoided the formation of thermally induced martensite during reversion annealing.
5. Hardness values were increased by increasing N in 201L stainless steel.

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