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# Effect of Cold Rolling on Microstructure and Mechanical Properties of AISI 304N Stainless Steel

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**Abstract.** Effect of cold rolling at room temperature on microstructure and mechanical properties of a nitrogen-bearing austenitic stainless steel AISI 304N was studied in this paper. The cold rolling reductions from 5% to 90% were adopted for the cold rolling test. Microstructures were observed by an optical microscopy, volume fractions of strain induced  $\alpha'$ -martensite were measured by a ferritescope, and deformation induced transformation was investigated by X-ray diffraction. Hardness and tensile tests were carried out to determine the mechanical properties. The results showed that with increase of cold rolling reduction, the volume fraction of strain induced  $\alpha'$ -martensite increased while grains were elongated along the rolling direction. The formation of  $\alpha'$ -martensite and the lamellar grains resulted in the significant strengthening of the steel and substantial decrease of elongation.

## 1. Introduction

Austenitic stainless steels (ASSs) are widely used in petroleum, chemical, nuclear power, transportation and other industrial and civil applications for their excellent corrosion resistance, formability, weldability and low temperature toughness [1]. However, yield strength of the annealed ASSs is relatively low which limits their application in some areas such as structures where require the steels have high strength [2]. There are several approaches to strengthen ASSs, such as solid solution strengthening, fine-grained strengthening, precipitation strengthening, work hardening, etc [3]. Generally, ASSs possess high strain hardening rate, so cold working is usually used to strengthen these steels [4]. Many cold working processes can significantly improve the yield strength of ASSs, such as equal channel angular processes, multidirectional forging, high-pressure torsion and cold rolling, etc. [5-8]. Among these cold working processes, cold rolling is the most efficient method for production of sizeable products and has the advantage of lower cost.

During cold rolling of ASSs, the variation of microstructure may include the formation of stacking faults, deformation bands,  $\epsilon$ -martensite, deformation twins and  $\alpha'$ -martensite, the multiplication of dislocation and evident grain subdivision, etc [9, 10]. These microstructure features increase the



difficulty of dislocation movement and thus strengthen the material. The microstructure evolution of ASSs during cold rolling is mainly depended on stacking fault energy (SFE), which is a function of chemical composition and temperature [11]. Allain et al. [12] reported that mechanical twins form when SFE is between 12 and 35mJ/m<sup>2</sup>, while strain induced martensite forms when SFE is below 18mJ/m<sup>2</sup>. Strain induced martensite is a unique feature of ASS [13], and it substantially improve the strength of ASS. In addition to SFE, the formation and amount of strain induced martensite also depend on Md30 (the temperature at which 50% martensite are transformed from austenite when true strain is 30%), initial austenite grain size, deformation degree, strain rate, etc [14, 15].

In recent years, nitrogen alloyed ASSs such as AISI 304N, AISI 316LN, etc, have drawn much attention for their excellent comprehensive properties [16-18]. Compared with AISI 304, AISI 304N possesses higher yield strength, better local corrosion resistance and low-temperature toughness, etc. It is a promising material to replace the traditional AISI 304 in many applications, such as transportation of the cryogenic materials like LNG (Liquefied Natural Gas). While the evolution of microstructure and mechanical properties of AISI 304 during cold rolling have been investigated in the previous works [19-21], no such studies have been reported on AISI 304N. In this paper, the effect of a wide range of cold rolling reductions (5%~90%) at room temperature on microstructure and mechanical properties of AISI 304N ASS was investigated.

## 2. Material and Methods

The specimen used in this study was the hot rolled and solution annealed AISI 304N ASS plate. The thickness of the plate was 5.0mm, and the manufacturing route of the plate was: 40t Electric Arc Furnace (EAF) → 40t Argon Oxygen Decarburization (AOD) furnace → Continuous Casting (CC) → 180 mm × 1280 mm slab → slab grinding → reheating → continuous hot rolling → coiling → continuous annealing and pickling. The chemical composition and Md30 temperature [14] of the specimen used in this study are listed in Table 1. Microstructure of the as-received sample is shown in Figure 1, the average grain size of the equiaxed austenite grain is about 45μm. There are many annealing twins in the metallograph but the precipitated carbonitride and δ ferrite cannot be observed. Specimens with the size of 5mm×150mm×500mm were cut from the plate. These specimens were unidirectionally cold rolled to different thicknesses (shown in Table 2) by a 4-high reversing cold rolling mill at room temperature with the rolling speed of 6 rpm. During cold rolling, the cold reduction for each pass was about 2%.

**Table 1.** Chemical composition (mass%) and Md30 temperature (°C) of the investigated steel.

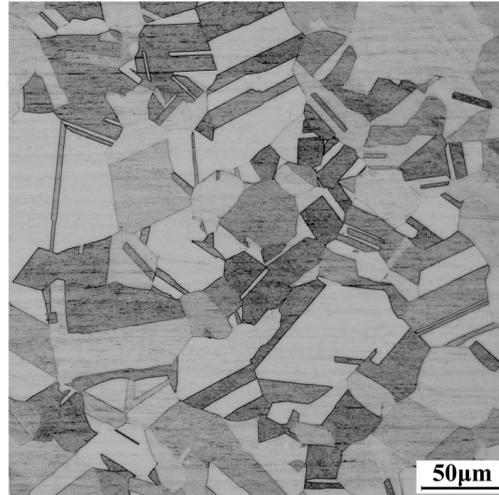
Type	C	Si	Mn	P	S	Cr	Ni	N	Fe	Md <sub>30</sub>
AISI 304N	0.04	0.39	1.10	0.027	0.001	19.08	8.07	0.127	Balance	-30.7

**Table 2.** AISI 304N specimens' cold rolling reduction and corresponding thickness.

Cold rolling reduction (%)	0	5	10	20	30	40	50	60	70	80	90
Thickness (mm)	5	4.75	4.5	4	3.5	3	2.5	2	1.5	1	0.5

Microstructures of the cold rolled specimens were analyzed on the cross section and along the rolling direction by an optical microscopy (LEICA DM4000 M LED). The etchant used to reveal the microstructure was composed of 70ml HCl + 100ml distilled water + 10g FeCl<sub>3</sub>. X-ray diffraction was carried out to reveal the phase components of the specimens with different cold rolling reductions. The X-ray studies were conducted using an X'Pert PRO X-ray diffractometer with Co K $\alpha$  radiation at 35kV and 50mA. A ferrite content measuring instrument (Fischer Technology Ferritescope, model FMP30) was used to measure the amount of ferromagnetic  $\alpha'$ -martensite phase. The device was calibrated by the standard samples before measuring and the results were adapted by multiplying the correlation factor of 1.7 [22]. Tensile tests were carried out with the cross-head speed of 2mm/min at room temperature by a testing machine (Z100). The tensile test specimens with the gauge length of 50mm

and the width of 12.5mm were machined. Tensile direction was parallel to the rolling direction. Vicker hardness tests were carried out with the load of 5kg load.



**Figure 1.** Optical micrograph of the as-received AISI 304N ASS specimen.

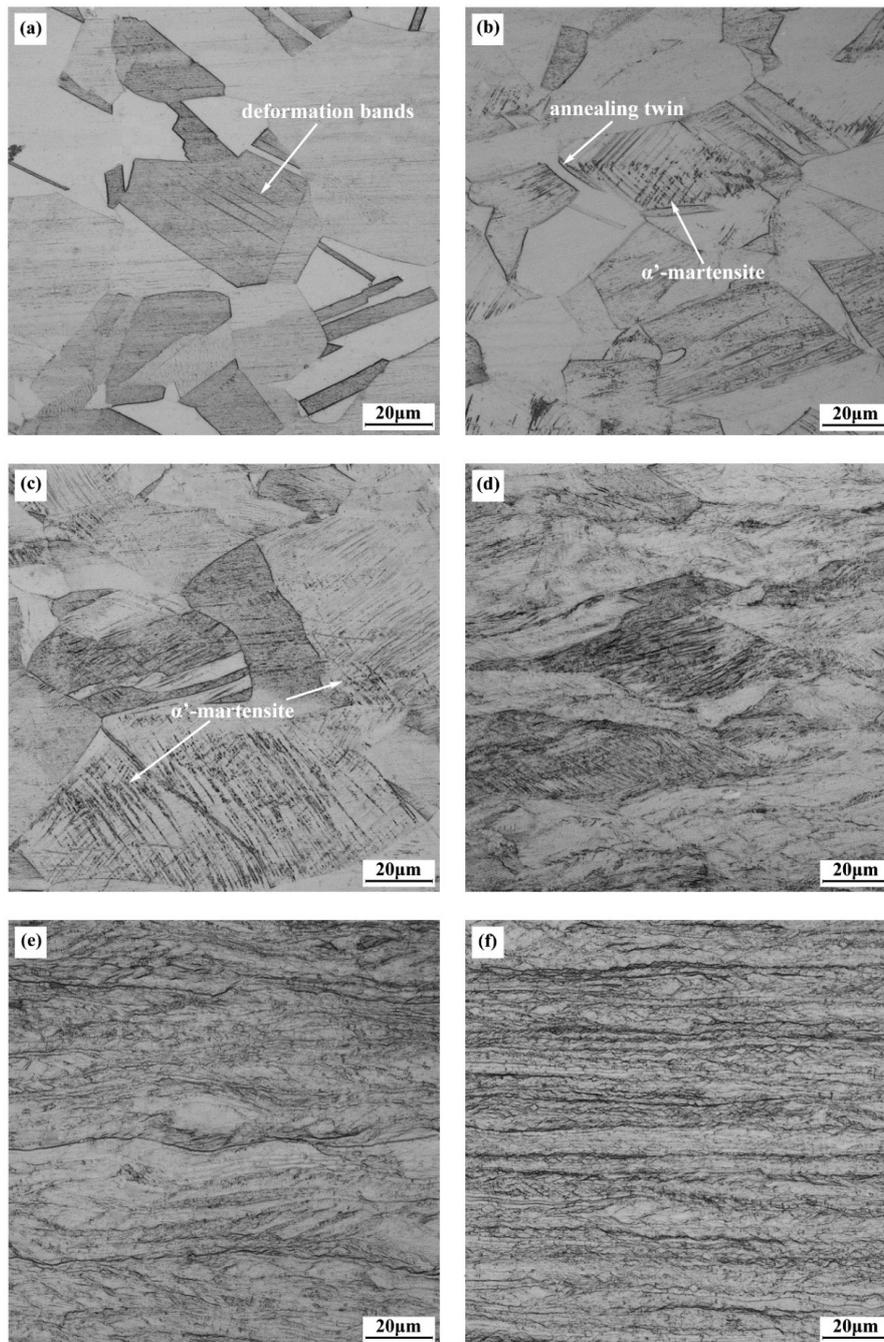
### 3. Results and discussion

#### 3.1. Microstructure

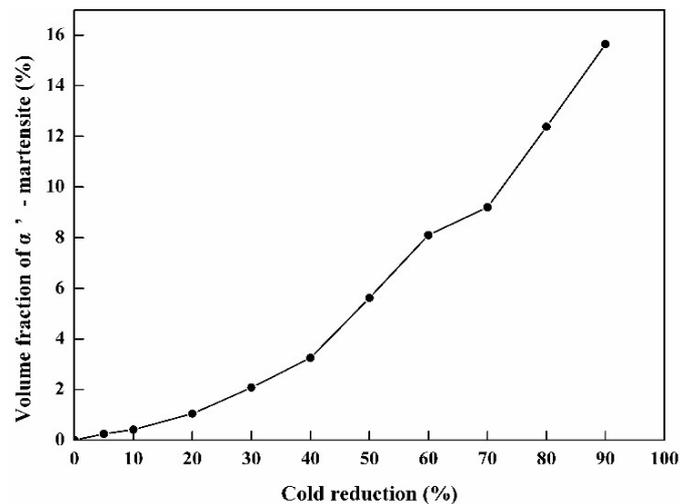
Figure 2 shows the optical micrographs of the cold rolled specimens of AISI 304N ASS with different cold rolling reductions. At the beginning of cold deformation, grain shape kept equiaxed, deformation bands can be observed in only a few grains (Figure 2(a)). It can be seen that deformation bands formed in one direction and parallel to each other which means only one slip system was activated on the (111) $\gamma$  plane at this stage. Deformation bands formed in more grains when cold rolling reduction reached to 20% (Figure 2(b)). In some grains deformation bands formed in different directions and crossed each other which means that more slip systems were activated in different (111) $\gamma$  planes. Deformation bands ended at grain boundaries and annealing twin boundaries meaning that grain boundaries and annealing twin boundaries prevent dislocation slip [23]. In addition, wavy bands can be observed, and some grains and annealing twins slightly lost their equiaxed state. Only a small amount of the magnetic  $\alpha'$ -martensite formed at this deformation reduction. And  $\alpha'$ -martensite often nucleated at the intersections of these deformation bands. When cold rolling reduction increased to 30% (Figure 2(c)), more grains were tilted along the rolling direction, and some annealing twins were severely distorted. The number of wavy and criss-crossing deformation bands increased, more  $\alpha'$ -martensite were nucleated and grew into the neighboring austenite matrix. Murr, et al. [24] found that  $\alpha'$ -martensite laths were formed by the stacking of many small  $\alpha'$ -martensite embryos. All grains tilted into the rolling direction and more  $\alpha'$ -martensite formed when cold reduction increased to 50% (Figure 2(d)). As cold rolling reduction went up to 70% and 90% (Figure 2(e) and (f)), the elongated grains became lamellar with high dislocation density and strain induced  $\alpha'$ -martensite continued to grow by consuming the austenite matrix [19].

Figure 3 shows the relationship between volume fractions of strain induced  $\alpha'$ -martensite and cold rolling reductions. It can be seen from Figure 3 that with the increase of cold reduction, volume fraction of  $\alpha'$ -martensite increases. Additionally, rate of  $\gamma \rightarrow \alpha'$  transformation is low at the early cold rolling reductions, and then it gradually increases with the increase of cold reduction. This phenomenon shows that at the early stage of cold deformation, instead of the increase of  $\alpha'$ -martensite fraction, nucleation sites for  $\alpha'$ -martensite were formed. These nucleation sites are usually located at the regions where there has large strain gradient, such as intersections of shear bands [25, 26]. The cold rolling reduction of 20% can be regarded as the critical cold rolling reduction at which  $\gamma$  started to

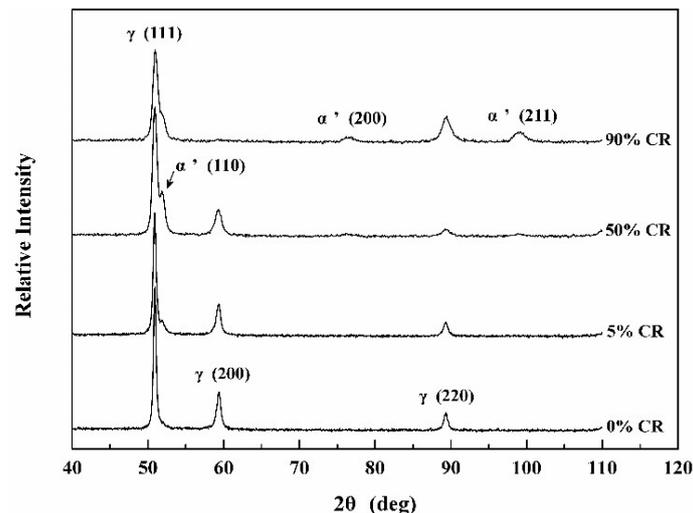
transform to  $\alpha'$ -martensite in this material. When cold rolling reductions reached to 80% and 90%, volume fractions of  $\alpha'$ -martensite got up to 12.4% and 15.6% respectively. From the transformation curve of strain induced  $\alpha'$ -martensite, it can be seen that the ability of the formation of strain induced  $\alpha'$ -martensite in 304N is much lower than that of 304 [10, 20, 27]. This phenomenon should be attributed to the difference in the chemical composition of these stainless steels, especially the difference of nitrogen content. Nitrogen can evidently increase the thermal and mechanical stabilities of ASSs and may increase the SFE of ASSs [28, 29], thus can effectively inhibit the formation and amount of strain induced martensite.



**Figure 2.** Optical micrographs of AISI 304N ASS with different cold rolling reductions: (a) 5%, (b) 20%, (c) 30%, (d) 50%, (e) 70% and (f) 90%.



**Figure 3.** The effect of cold rolling on the volume fraction of  $\alpha'$ -martensite in AISI 304N ASS.



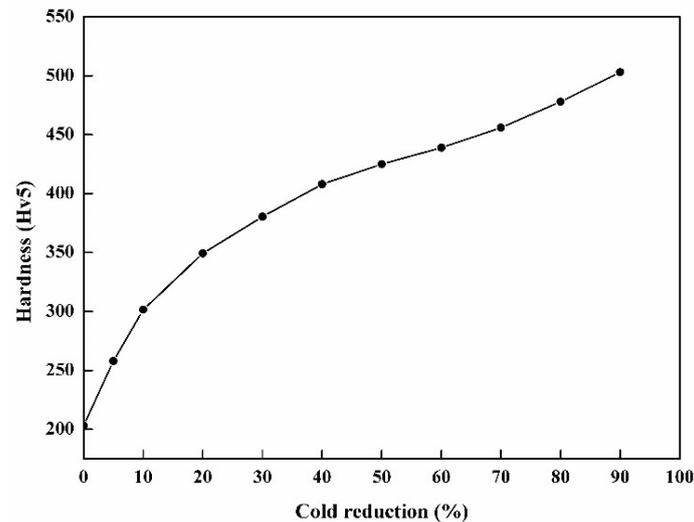
**Figure 4.** X-ray diffraction patterns of the as-received and cold rolled AISI 304N ASS specimens.

Figure 4 shows the X-ray diffraction patterns of AISI 304N ASS cold rolled at room temperature to different thicknesses. It can be seen that the microstructure of the as-received sample was fully austenitic. With the increase of cold rolling reduction from 5% to 90%, the diffraction intensity of  $\gamma(200)$  and  $\gamma(111)$  gradually decreased while that of  $\alpha'(200)$  and  $\alpha'(211)$  began to appear at about 50% cold rolling reduction and then grew slowly. This means that during cold rolling of AISI 304N ASS, the austenite phase gradually decreased and transformed into  $\alpha'$ -martensite phase. When cold rolling reduction reached to 90%, the main part of the microstructure was still austenite, while there also existed some  $\alpha'$ -martensite.

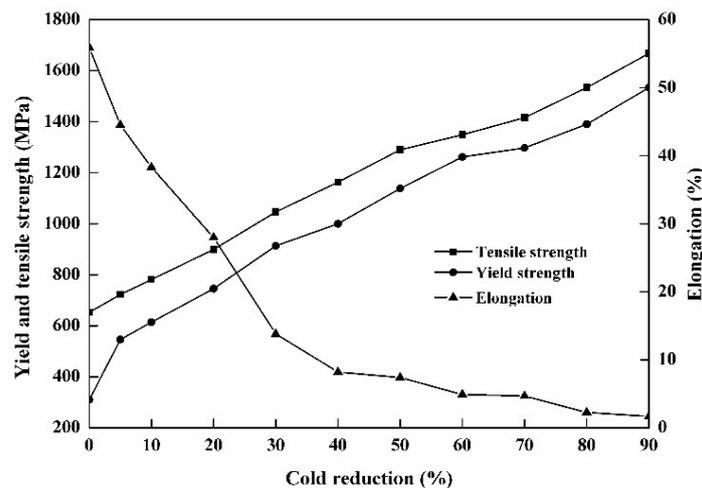
### 3.2. Mechanical properties

The variation of hardness with cold rolling reduction is shown in Figure 5. It can be seen that cold rolling had greatly hardened the material. Additionally, the strain hardening effect is much more evidently when cold rolling reduction is smaller than 40%, at which the hardness increased about 100% as that of the solution annealed state. However, when cold rolling reduction went up from 40% to 90%, the hardness only increased about 23%. The main reasons for the increase of hardness may include the formation of mechanical twins, increase of dislocation density and the nucleation and

growth of strain induced  $\alpha'$ -martensite [4]. The maximum Vickers hardness that AISI 304N ASS reached in this paper is 503.



**Figure 5.** The effect of cold rolling on hardness of AISI 304N ASS.



**Figure 6.** The effect of cold rolling on yield strength, tensile strength and elongation of AISI 304N ASS.

Figure 6 shows the effect of cold rolling on yield strength, tensile strength and elongation of AISI 304N ASS. Figure 6 indicates that cold rolling considerably increases the yield and tensile strengths while substantially decreases the elongation of AISI 304N ASS. When cold rolling reduction reached up to 90%, the yield and tensile strengths of the solution annealed state significantly increased from 310MPa and 653MPa to 1534MPa and 1668MPa respectively. On the contrary, the elongation decreased drastically from 56% to only 1.5%.

It can be seen from Figure 3 and Figure 4 that the phase of AISI 304N ASS changed from the single austenite in the solution annealed state to the dual phase of austenite +  $\alpha'$ -martensite with the increase of cold rolling reduction. For this dual phase structure, the following equation [30] can be used to calculate the overall yield strength:

$$\sigma_{0.2} = F_A \sigma_{0.2} (\text{Austenite}) + F_M \sigma_{0.2} (\text{Martensite}) \quad (1)$$

where FA and  $\sigma_{0.2}$  (Austenite) are the volume fraction and yield strength of austenite while FM and  $\sigma_{0.2}$  (Martensite) are the volume fraction and yield strength of martensite. Meanwhile, with increase of cold rolling reduction, the dislocation density of austenite and  $\alpha'$ -martensite increased while the grain size of the two phases was getting smaller. Generally, the yield strength of austenite or martensite phase can be expressed by a modified Hall-Petch-type relationship [25]:

$$\sigma_{0.2} = \sigma_0 + K_e D^{-0.5} + \alpha G b \rho^{0.5} \quad (2)$$

where  $\sigma_0$  is the yield strength of dislocation-free single crystalline phase, D is the mean grain size, G is the shear modulus, b is the Burgers vector,  $\rho$  is the dislocation density, and  $K_e$  and  $\alpha$  are constants. Based on equations (1) and (2), it is reasonable that strength increases with the increase of cold rolling reduction. On the other hand, with increase of cold rolling reduction, the deformation capacity of remained austenite decreases [31], and deformation capacity of strain induced  $\alpha'$ -martensite is much lower than that of austenite, so elongation decreases with increase of cold rolling reduction.

#### 4. Conclusion

1) Microstructure of the solution annealed state of AISI 304N ASS was fully austenitic. With increase of cold rolling reduction,  $\alpha'$ -martensite nucleated at intersections of shears bands and gradually grew bigger, while dislocation density increased and grains became elongated.

2) The volume fraction of  $\alpha'$ -martensite was 15.6% when cold rolling reduction reached up to 90%. Compared with the conventional AISI 304 ASS, the ability of the formation of strain induced  $\alpha'$ -martensite in AISI 304N is much lower.

3) The formation and growth of  $\alpha'$ -martensite, grain refinement and multiplication of dislocation during cold rolling led to significant strengthening. The yield strength increased from 310 MPa in the solution annealed state to 1534 MPa after cold rolling to a total cold rolling reduction of 90%.

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