



On the strengthening mechanisms of high nitrogen austenitic stainless steels

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ABSTRACT

The effect of microstructure and dispersion of secondary phases on tensile mechanical properties of two high nitrogen austenitic stainless steels subjected to annealing or rolling at 1000 °C was studied. The solid solution strengthening gave the main contribution to overall strength after annealing. Similar strength contribution was provided by the dislocation density after rolling that resulted in almost twofold increase in the yield strength.

1. Introduction

High nitrogen austenitic stainless steels are used for various engineering applications as structural materials due to beneficial combination of high strength, excellent ductility, toughness, and corrosion resistance [1–3]. Additions of nitrogen may provide an almost twofold increase in the strength in comparison with conventional austenitic steels [1–3]. The effect of the nitrogen content on the strength was attributed to the solid solution strengthening and the nitrogen-enhanced austenite grain strengthening [3–5]. Several studies dealt with the nitrogen effect on the strength of austenitic steels in annealed conditions [1,3,4,6,7]. The Hall-Petch coefficient and the dislocation strengthening factor have been clarified for the high nitrogen steels with recrystallized microstructure. However, the strengthening mechanisms for high nitrogen austenitic steels in the work hardened condition have not been fully investigated in spite of frequent usage of cold-to-hot rolling for producing the steel semi-products. The aim of the present paper, therefore, is to consider the strengthening mechanisms in high nitrogen steels in the annealed or hot worked conditions.

2. Materials and methods

Two austenitic stainless steels with different N and C contents denoted here as Steel 1 (Fe-0.05C-0.72 N-20.74Cr-8.08Ni-15.33Mn-1.04Mo-0.22 V-0.11Si-0.005S-0.005P), and Steel 2 (Fe-0.03C-0.62 N-20.0Cr-7.80Ni-14.8Mn-0.83Mo-0.20 V-0.20Si-0.008S-0.010P (all in wt. %)) were investigated. Steel 1 was obtained by plasma-arc remelting and

hot forging. Steel 2 was obtained as a laboratory metal (cast plate 40 mm thick), which was then annealed and hot-formed into a bar. Both steels were annealed at 1200 °C for 1 h followed by water quenching. A part of the steel samples was subjected to annealing at 1000 °C for 1 h followed by air cooling. Another part of the steel samples was hot rolled at 1000 °C to a total reduction of 60 % followed by water cooling.

The structural investigations were performed using a Quanta 600 scanning electron microscope with an electron back-scatter diffraction (EBSD) analyzer and a JEM-2100 transmission electron microscope (TEM). The grain size was evaluated using TSL OIM Analysis 6 software. The dislocation density was measured by counting the individual dislocations inside the grains/subgrains revealed by TEM. The volume fractions of particles were evaluated by using ThermoCalc (TCFE7 database). The tensile specimens with a gauge length of 12 mm and a cross-section of 3×1.5 mm² were tensioned at room temperature using an Instron 5882 testing machine. The shear modulus was measured using a resonant frequency damping analyzer RFDA Basic – IMCE NV.

3. Results and discussion

Typical microstructures that develop after annealing or rolling are shown in Fig. 1. The microstructural parameters and the tensile properties of the steels are summarized in Table 1. Annealing resulted in the uniform recrystallized austenitic microstructures with low dislocation densities. The large fractions of annealing twin boundaries comprising 0.4 and 0.6 in Steel 1 and Steel 2, respectively, are indicative of low stacking fault energy that should suppress the dislocation

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rearrangement. Hot rolling led to the elongation of initial grains along the rolling direction (RD) and the formation of partially recrystallized microstructure with micron scale grains. An average grain size decreased by a factor of 10 and the dislocation density increased by a factor of 10^2 in the both steels subjected to hot rolling (Table 1). The formation of Cr_2N nitrides took place in both steels under annealing (Fig. 2a and b). These carbides exhibit primarily an oval shape in Steel 1. In Steel 2, the coarse Cr_2N nitrides with round shape coexist with relatively fine carbides with plate-like shape. The Cr_2N nitrides that are observed in the steels after rolling are finer than after annealing (Fig. 2c and d). In addition, hot rolling induced the precipitation of V(C,N) carbonitrides with round shape (Fig. 2c and d). The precipitation of these particles during hot rolling might be promoted by the high dislocation densities.

The engineering stress-elongation curves after annealing and after hot rolling are shown in Fig. 3a. The annealed steels exhibited pronounced strain hardening stage with a ratio of the yield strength to the ultimate tensile strength (YS/UTS) of 0.55. Hot rolling shortened the strain hardening stage and increased YS/UTS ratio up to 0.9. The yield strength of Steel 1 is slightly higher than that of Steel 2 (Table 1) due to higher nitrogen content in the annealed conditions. Hot rolling increased YS by a factor of 2, while UTS increased by about 40 % in the both steels. It is worth noting that YS and UTS of two steels became nearly the same after hot rolling. Therefore, an increase in the dislocation density diminished the effect of nitrogen on the steel strength.

The yield strength of the present steels can be estimated using a linear combination of the strengthening mechanisms that can be written as follows [8,9],

$$YS = \sigma_0 + \sigma_{SS} + \sigma_{part} + \sigma_{HP} + \sigma_{disl} \quad (1)$$

where σ_0 is the lattice friction stress of about 64 MPa in high purity austenite [1], σ_{SS} is the solid solution strengthening, σ_{part} is the precipitation strengthening, σ_{HP} is the grain boundary strengthening, and σ_{disl} is the dislocation strengthening. The solid solution strengthening

Table 1

Characteristics of the microstructure and mechanical properties of high-nitrogen austenitic stainless steels subjected to annealing or rolling at 1000 °C.

	Steel 1		Steel 2	
	Annealing at 1000 °C		Rolling at 1000 °C	
Average grain size, D (μm)	100 ± 20	90 ± 20	9 ± 2	7 ± 2
Dislocation density, ρ (m ⁻²)	4.8 × 10 ¹² ± 0.5	4.4 × 10 ¹² ± 0.5	5.5 × 10 ¹⁴ ± 0.5	5.8 × 10 ¹⁴ ± 0.5
Volume fraction of particles, F	Cr_2N	0.033	0.024	0.033
	V(C, N)	–	–	0.003
Average size of particles, d (nm)	Cr_2N	260 ± 30	180 ± 30	190 ± 30
	V(C, N)	–	–	35 ± 5
Yield strength, YS (MPa)	500 ± 10	460 ± 10	1060 ± 10	1060 ± 10
Ultimate tensile strength, UTS (MPa)	870 ± 10	840 ± 10	1200 ± 10	1170 ± 10
Total elongation, δ (%)	64 ± 2	58 ± 2	21 ± 2	17 ± 2

(σ_{SS}) can be expressed by the modified Irvine equation [10],

$$\sigma_{SS}(\text{annealing}) = 496 \times (\%N - 0.25) + 20.1 \times (\%Si) + 3.7 \times (\%Cr - 1.5) + 14.6 \times (\%Mo) + 18.6 \times (\%V) \quad (2)$$

$$\sigma_{SS}(\text{rolling}) = 496 \times (\%N - 0.25) + 20.1 \times (\%Si) + 3.7 \times (\%Cr - 1.5) + 14.6 \times (\%Mo) \quad (3)$$

According to ThermoCalc, the precipitation of V(C,N) carbonitrides and Cr_2N nitrides consumes about 0.25 % N, 1.5 % Cr, whole C and V content in the present steels. Nevertheless, the content of N provides the main contribution to the solid solution strengthening. The precipitation strengthening (σ_{part}) can be expressed by the Ashby-Orowan equation

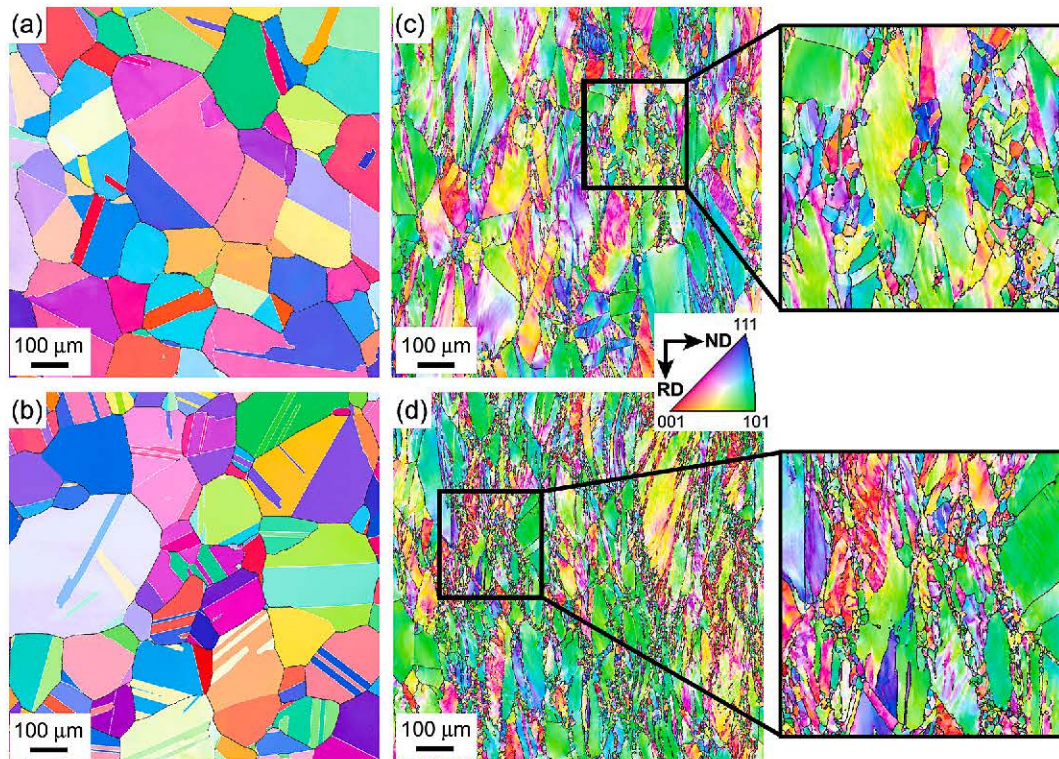


Fig. 1. Microstructures of Steel 1 (a, c) and Steel 2 (b, d) steels after annealing (a, b) or rolling (c, d) at 1000 °C. The high-angle boundaries and the twin boundaries are indicated by the black and white lines, respectively.

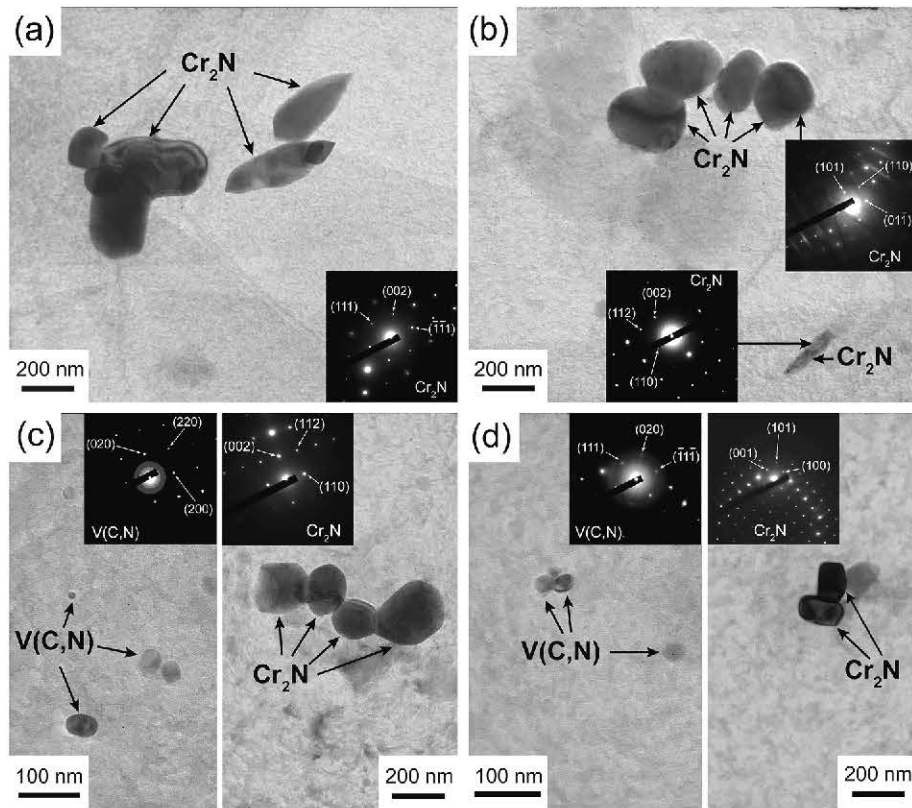


Fig. 2. TEM images of secondary phase particles on carbon replicas of Steel 1 (a, c) and Steel 2 (b, d) steels after annealing (a, b) or rolling (c, d) at 1000 °C.

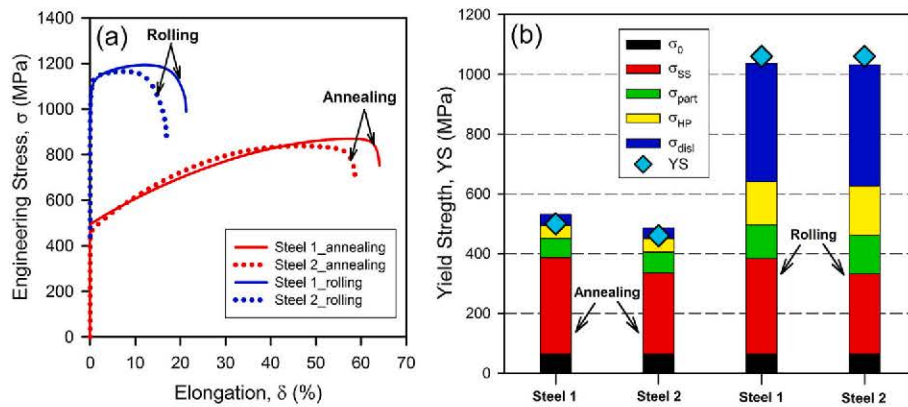


Fig. 3. Engineering stress – elongation curves (a) and contributions of different strengthening mechanisms to the yield strength (b) of high-nitrogen austenitic stainless steels after annealing or rolling at 1000 °C.

[11],

$$\sigma_{part} = 0.84kM \frac{Gb}{2\pi(L-d)} \ln\left(\frac{d}{4b}\right) \quad (4)$$

where G is the shear modulus (experimentally measured as 75 GPa for the present steels), b is the Burgers vector, d is the particle size (Table 1), M is the Taylor factor, and $k = 1.2$ is a constant [11]. The center-to-center interparticle spacing, L , is given by [12],

$$L = \left(\frac{6F_v}{\pi d^2}\right)^{-0.5} \quad (5)$$

where F_v is the volume fraction of particles (Table 1). The precipitation strengthening originated from different types of precipitates was calculated as $\sigma_{part} = (\sigma_{part1}^2 + \sigma_{part2}^2)^{0.5}$ [13]. The grain boundary

strengthening (σ_{HP}) can be evaluated by the Hall-Petch-type relationship [14,15],

$$\sigma_{HP} = K_{HP}D^{-0.5} \quad (6)$$

where K_{HP} is a Hall-Petch coefficient. $K_{HP} = 435 \text{ MPa} \times \mu\text{m}^{0.5}$ reported for austenitic steel [16] was taken for the present calculations. It should be noted that a number of annealing twins including non-symmetric $\Sigma 3$ CSL boundaries, which possess the different crystallography and properties [17–19], is apparently smaller in Steel 1 (Fig. 1a and b). Therefore, the grain boundary strengthening after annealing evaluated by Eq. (6) may be comparatively overestimated for Steel 1.

The dislocation strengthening (σ_{disl}) due to the high dislocation density (ρ) can be expressed by Taylor – type relationship [20],

$$\sigma_{disl} = \alpha M G b \rho^{0.5} \quad (7)$$

where α is a dislocation strengthening factor of about 0.3 for austenitic steels [21].

The variation of the strengthening contributors in accordance with Eq. (1) is shown in Fig. 3b along with experimental YS. The solid solution strengthening provides the main contribution to the strength after annealing, so the yield strength of Steel 1 is higher by 40 MPa than that of Steel 2. The dislocation strengthening exceeds the solid solution strengthening after hot rolling. Therefore, both the solid solution and the dislocation density are the major strengtheners after hot rolling. The contribution of grain boundary strengthening to the yield strength is insignificant, since the grain size is rather large after hot rolling. The contribution of the precipitation strengthening is also relatively small, since the Cr₂N nitrides have large dimensions, while the fraction of fine carbonitrides is small.

4. Summary

The strengthening mechanisms were clarified for two austenitic stainless steels with different N and C contents subjected to annealing or rolling at 1000 °C. In the annealed conditions, the steel with higher N content is characterized by higher yield strength owing to the solid solution strengthening. Hot rolling increased the dislocation density, which became primary strengthener and doubled the yield strength. Both Steel 1 (0.72 %N-0.05 %C) and Steel 2 (0.62 %N-0.03 %C) exhibit the same yield strength of 1060 MPa after hot rolling irrespective of different N content.

CRediT authorship contribution statement

M.V. Odnobokova: Investigation, Data curation, Writing – original draft. **A.N. Belyakov:** Data curation, Writing – original draft. **P.D. Dolzhenko:** Investigation. **M.V. Kostina:** Formal analysis, Writing – review & editing. **R.O. Kaibyshev:** Formal analysis, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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References

- [1] V.G. Gavriljuk, H. Berns, *High Nitrogen Steels Structure, Properties, Manufacture, Applications*, Springer, Germany, 1999.
- [2] K.H. Lo, C.H. Shek, J.K.L. Lai, *Mater. Sci. Eng. R* 65 (2009) 39–104.
- [3] A. Sisev, S. Tsimerman, M. Kostina, A. Il'inskii, M. Perkas, L. Rigina. *Rus. Met. (Metally)*. 2018. (2018). 545–551.
- [4] J.W. Simmons, *Mater. Sci. Eng. A* 207 (1996) 159–169.
- [5] M.L.G. Byrives, M. Grujicic, W.S. Owen, *Acta Metall.* 35 (1987) 1853–1862.
- [6] V. Ganesan, M.D. Mathew, K.B. Sankara Rao, *Mater. Sci. Technol.* 25 (2009) 614–618.
- [7] H. Berns, V. Gavriljuk, S. Riedner, *High Interstitial Austenitic Stainless Steels*, Springer, Germany, 2013.
- [8] T. Gladman, F.B. Pickering, T.N. Baker (Eds.), *Yield, Flow and Fracture of Polycrystals*, Applied Science Publishers, London, UK, 1983, pp. 141–198.
- [9] Z. Xiong, I. Timokhina, E. Pereloma, *Progr. Mater. Sci.* 118 (2021), 100764.
- [10] K. Irvine, *J. Iron Steel Inst.* 207 (1969) 1017–1028.
- [11] M.F. Ashby, On the Orowan Stress, in: A.S. Argon (Ed.), *Physics of Strength and Plasticity*, M.I.T. Press, Cambridge, 1969, pp. 113–131.
- [12] F.J. Humphreys, M. Hatherly, *Recrystallization and related annealing phenomena*, 2nd ed., Elsevier, UK, 2004.
- [13] T. Koppelaar, D. Kuhlmann-Wilsdorf, *Appl. Phys. Lett.* 4 (1964) 59–61.
- [14] E. Hall, *Proc. Phys. Soc. Sect. B* 64 (1951) 742.
- [15] N. Petch, *J. Iron Steel Inst.* 174 (1953) 25–28.
- [16] Z. Yanushkevich, A. Belyakov, R. Kaibyshev, C. Haase, D.A. Molodov, *Mater. Character.* 112 (2016) 180–187.
- [17] F. Ernst, M.W. Finnis, A. Koch, C. Schmidt, B. Straumal, W. Gust, *Z. Metallkd.* 87 (1996) 911–922.
- [18] V.G. Sursaeva, B.B. Straumal, A.S. Gornakova, L.S. Shvindlerman, G. Gottstein, *Acta Mater.* 56 (2008) 2728–2734.
- [19] B.B. Straumal, O.A. Kogtenkova, A.S. Gornakova, V.G. Sursaeva, B. Baretzky, *J. Mater. Sci.* 51 (2016) 382–404.
- [20] G.I. Taylor, *Proc. R. Soc. Lond. Ser. Contain. Pap. Math. Phys. Character.* 145 (1934) 362–387.
- [21] M.M. Abramova, N.A. Enikeev, R.Z. Valiev, A. Etienne, B. Radiguet, Y. Ivanisenko, X. Sauvage, *Mater. Lett.* 136 (2014) 349–352.