Effect of cold rolling on tensile properties and microstructure of high nitrogen alloyed austenitic steel

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The cold rolling strain effect on the tensile properties of a high nitrogen alloyed austenitic steel was systemically investigated. Quasi-static tensile experiments were performed on the samples with different cold rolling strain. The material possessed good balance between strength and ductility in the entire rolling strain range. Mechanical property and microstructure of the nitrogen alloyed austenitic steel were greatly affected by cold rolling strain. With the increase in cold rolling strain, the strength increased sharply but the ductility declined. That is related to the gradual changes of microstructure induced by the cold rolling process. The cold rolling process leads to substantial microstructural change, from the appearance of slip bands and twins at low cold rolling strain; the microtwins formation at intermediate cold rolling strain and followed by sequences of their bending, breaking and disappearance; and finally to the formation of drossy twins at the high cold rolling strain.

Keywords: Nitrogen alloyed austenitic steel, Cold rolling, Tensile behaviour, Microstructure, Twinning

Introduction

High nitrogen austenitic stainless steels (HNSS) are of potential significance for structural applications as they possess many appealing properties, such as high strength and ductility, desirable fracture toughness, adequate workhardening ability and good corrosion resistance.¹⁻⁵ These attractive properties are heavily related to solid solution nitrogen. Interstitial nitrogen is very effective on solid solution strengthening⁶ and stabilising austenitic structure.⁷ Therefore, phase transformation hardly happens in HNSS even after severe cold deformation even at cryogenic temperature.⁷⁻⁸ The mechanical properties and microstructure evolution, including stacking fault and stacking fault energy (SFE) of HNSS, were intensively researched.¹⁻³⁻⁹ Owing to the substitution of Ni by N, HNSS also can be used instead of more expensive Ni base austenitic stainless steels.¹⁰ N addition is a major factor to enhance tensile strength, which is also the main driving force for the intensive studies on HNSS in the past decades.¹¹⁻¹⁴

Grain size refinement can effectively improve the strength of metals and alloys according to the Hall–Petch relation.¹⁵ Cold rolling, during which severe plastic deformation can be generated, is a feasible and effective way to refine grain size.¹⁶⁻¹⁷ Moreover, recent investigations concerning the cold rolling of HNSS revealed the formation of deformation twins and slip lines.⁵⁻¹⁸ The microtwins can block dislocation motion and thus induce the increase in strength. Based on these results, we can envisage obtaining an HNSS with higher stress by cold deformation, especially considering the fact that HNSS exhibit excellent workhardening ability by the introduction of N. However, the relevant literatures are still quite limited.

In the present work, the mechanical properties of HNSS under a board cold rolling strain range were investigated by tensile tests. The evolutions of the microstructure were also detected. The unique tensile properties of HNSS after rolling were elucidated based on the cold rolling induced microstructure.

Experimental

Materials

The material used in the present study is an HNSS produced by Changchun University of Technology. The as received ingots are reported with the nominal chemical compositions of Fe-0.02C–22Cr–17Mn–2.43Mo–0.83N–0.27Si–0.21Nb–0.012P–0.004S (wt.%). The ingots were initially solution heat treated at 1150°C for 8 h followed by water quenching in order to keep all nitrogen in solid solution, eliminate the effect of casting hot forming and form a single austenitic equiaxed grain. The microstructure of the as received sample is the uniform coarse grain with grain size of about 20–55 μm. The as received material was supplied in plates of 20 mm thickness. Before mechanical treatments by cold rolling, the material was machined to obtain the desirable thickness with parallel faces. The rolling was carried out on a laboratory
two-high mill. The mill was set to give \( \approx 0.2 \text{ mm} \) thickness reduction per pass. Starting from plates with different initial thicknesses, rolling led after the last pass to sheets \( \approx 1.5 \text{ mm} \) thick. Sheets with 10, 30, 50 and 70% cold rolling strain were selected for the mechanical and structural analyses.

**Mechanical testing**

All dog bone shaped tensile specimens with a gauge cross-section of \( \approx 2.5 \times 0.9 \text{ mm} \) and a gauge length 8.0 mm were cut from the HNSS sheets using an electrodischarging machine and then were polished to mirror-like finish surface using SiC papers and 0.25 \( \mu \text{m} \) diamond suspension. Tensile tests (at least three tests for each cold rolling strain) were carried out on the MTS-810 system with a strain rate \( \varepsilon \) of \( 10^{-3} \text{ s}^{-1} \) at room temperature (RT).

**Microstructure investigation**

The crystallographic structure of the HNSS was determined by X-ray diffraction (D/max2500PC), which was also carried out by means of electron backscattering diffraction (EBSD) method using field emission scanning electron microscopy (FESEM, SUPRA-40). The data obtained by EBSD method were analysed by orientation imaging microscopy. The discrete step size used for EBSD mapping ranged from 10 to 100 nm. The morphologies of the fracture surface of the samples were examined by FESEM. The microstructures of the samples were observed using transmission electron microscope (TEM, JEM-2100F) under an accelerating voltage of 200 kV. Foil samples for EBSD and TEM observation were prepared by ion beam thinning using the Ion Polishing System (Gantan 691, USA) with voltage of 5 kV and tilt angle of 4–8°.

**Results**

**Tensile properties**

Figure 1 shows the engineering stress–strain curves of the samples cold rolled to 0, 10, 30, 50 and 70% strain, which is performed at a strain rate of \( 10^{-3} \text{ s}^{-1} \) and at RT. The HNSS shows the ultimate tensile strength \( R_m \) of 1001–2236 MPa and the yield strength \( R_{p0.2} \) of 589–1885 MPa with the fracture ductility \( A \) of 12.3–86.6% and uniform plastic strain \( U \) of 5.9–64.1%. It can be seen that, with increasing cold rolling strain, \( R_m \) and \( R_{p0.2} \) increase and \( A \) and \( U \) decrease. As reported by Fréchard et al., the energy absorption capacity of HNSS decreases with increasing warm rolling strain. As shown in Fig. 1, our HNSS shows a similar character that the as received material undergoes large strains before fracture and exhibits the largest area under the stress–strain curve and therefore possesses the highest energy absorption capacity; with increasing cold rolling strain, this energy absorption decreases quickly.

Figure 2a and b presents the variation of \( R_m \) and \( A \) with rolling strain for nitrogenous stainless steels including the literature data\(^{19-24}\) respectively. Despite some inconsistency in the absolute values for different nitrogen contents or different rolling techniques, a consistent trend is clear: \( R_m \) increase linearly with increasing rolling strain in the entire rolling strain range, and \( A \) decreases obviously from 0% rolling strain to 50% rolling strain, while a slight decreasing appears when the rolling strain is beyond 50%. It is worth noting that, in the entire rolling strain range, our HNSS has high strength and satisfactory ductility. As the rolling strain increases to 70%, the \( R_m \) of our HNSS increases to 2236 MPa; such high \( R_m \) value for HNSS has not been reported.
Microstructure

X-ray diffraction investigations
Deformation induced austenite–martensite transformation is well known in steels.$^{25,26}$ The X-ray diffraction patterns in Fig. 3 reveal that the structures of the HNSSs with different cold rolling strain are (face centred cubic) austenitic phase, and no characteristic peaks of other phases can be detected. In addition, the diffraction peaks of cold rolled samples are broader than the as received sample (0% cold rolling strain). Broadened diffraction peaks suggest grain refinement and/or increase in atomic level strain.

Electron backscattering diffraction investigations
The inverse pole figure colour maps of EBSD in Fig. 4 show the orientation change of samples before and after cold rolling deformation, which depend on their crystallographic orientation components. The crystallographic orientations on the observed surface are displayed by the colours shown in the stereographic triangle superimposed in Fig. 4a. The microstructure of the sample with 0% cold rolling strain was composed of uniformly equiaxed grains with random crystalline orientations and predominant high angle GBs. With the increasing cold rolling strain, the crystal orientation of the transverse direction rotated, and the trace of orientation change moved towards the primary slip direction, as shown in Fig. 4b–e. Each grain surrounded by different colours corresponds to a eutectoid block, which is defined as an area where the austenite has an identical crystallographic orientation. Compared with the austenite grain size (40 μm) of 0% cold rolling strain, the grain size becomes finer with increasing cold rolling strain. High angle boundaries (15° ≤ misorientation gap) and low angle boundaries (2° ≤ misorientation gap <15°) can be seen in the Fig. 4c–e respectively. There are many low angle boundaries in one block, which is due to the existence of colonies within the block.

As illustrated in Fig. 4a, twins are observed in the sample with 0% cold rolling strain, which probably formed during the solution heat treatment process. With increasing cold rolling strain, more twins or microtwins are appeared, as shown in Fig. 4b and c, which probably formed during the cold rolling process. However, at high cold rolling strain (50 and 70%), the microstructures in EBSD maps are not clear. Therefore, the TEM images were given in the next section.

Transmission electron microscope investigations
As shown in Fig. 5a, the morphology of the sample with 0% cold rolling strain has a low dislocation density. Figure 5b displays a few dislocations in coplanar bands. The latter are piled up at twin boundaries and/or slip bands. The TEM images in Fig. 5c–e show the microstructure of the samples cold rolled to 30, 50 and 70% reduction respectively. Under these cold rolling conditions, the dislocation density is very high. With increasing cold rolling strain, the microtwins density increases. For the sample cold rolled to 30% reduction, a number of mechanical microtwins with parallel interfaces are found in the austenitic grains (Fig. 5c). The derived twin plane is clearly identified as $\{\overline{1}1\}$ plane. At high cold rolling strain (50%), the interfaces of twins are bent and broken (Fig. 5d). When cold rolling reduction
increases up to 70%, the longer twin interfaces disappear, which changes to a lot of shorter twin interfaces with high dislocation density (Fig. 5e). Figure 5f is the local enlarged area of location of white arrowhead in e. Figure 5a–e, the width of twins decreases with increasing cold rolling strain. As the strain goes up to 30%, nanotwin structures (the width of twin is \(<100 \text{ nm}\)) are found. As the strain goes up to 50%, the width of twins is further decreasing, and some nanograins are formed. As the strain up to 70%, a large number of nanotwins are broken, and the minimum width of twin is only 3–5 nm.

Field emission SEM investigations

For further illuminating the different strength and ductility trend at different cold rolling strains, the FESEM fracture morphologies of the HNSSs are shown in Fig. 6. From these figures, it can be clearly seen that many deformation bands were observed on the profile of the fracture samples. These deformation bandings mean that local plastic deformation has occurred. With increasing cold rolling strain, the deformation bandings become more uniform, which demonstrates that the location deformation of samples at high cold rolling strains was greatly weakened, which could improve the strength. Furthermore, there are many microcracks within these bands as marked by white cirques in Fig. 6. It should be noted that, with increasing cold rolling strain, the edges of microcracks become smoother, which could restrain microcracks to propagate effectively. Recent analysis pointed to the fact that the post-uniform elongation is believed to be caused by the propagation of microcracks.31 Therefore, post-uniform deformation of samples at high cold rolling strains was delayed, and this improves the post-uniform plastic strain and the ductility.

Discussion

The present investigation is focused on the effect of cold rolling on the tensile properties and microstructure evolution of HNSS. Compared with classical stainless steels,19–24 the HNSS used in the present study exhibits a much higher strength and satisfactory ductility, which is attributed to several factors, including chemical composition, grain size and interstitial nitrogen content. The nitrogen strength is due to the misfit between the interstitial nitrogen atoms and the octahedral lattice voids, leading to strains in the surrounding lattice.1,28 Furthermore, nitrogen is known to increase shear stress due to its interaction with dislocations.29 The yield strength is also be reinforced by grain size refinement.30 A close relationship was observed between the mechanical behaviour and the microstructural development during cold rolling process.

The as received HNSS exhibits good strength and ductility, which is due to the twins formed during the solution heat treatment process. The ultimate strength obviously increased as cold rolling to 10% strain. This should be relative to the increase in dislocation density in the sliding process (Fig. 5b). In addition, dislocation movement can also be restricted by the interstitial nitrogen atoms. For 30% cold rolling strain, fine subgrain or microtwin features can be observed in the original austenite grains, as seen in the inverse pole figure colour maps in Fig. 4c, as well as in the TEM images in Fig. 5c. The density of microtwins also increases with rolling strain. The occurrence of high density of twins is a consequence of low SFE of austenitic stainless steels.31 It should be noted that the effect of nitrogen on the SFE depends on the component. In Cr–Ni steels, the SFE decreases at small additions of nitrogen and increases at higher nitrogen contents. In Cr–Mn steels, the SFE adopts an opposite behaviour: it first increases and then decreases with increasing nitrogen concentration.29 Our HNSS is sort of Cr–Mn steel; thus, it has low SFE and trend to form twins. As the strain goes up to 50%, twins are bent and broken, and the widths of twins are far \(<100 \text{ nm}\) (Fig. 5d). Further deformation up to 70% strain leads to the fraction of twin structures and high dislocation density (Fig. 5e). The deformation by cold rolling starts...
by early slip and then twinning up to 50% rolling strain. At last, these twins fractured at large rolling strain (70%), and some nanograins were formed. The slip and twinning mechanisms should be concomitant during the early deformation. The microtwins serving as barriers to dislocation, same like grain boundary, lead to an increase in strength. In fact, twin lamellar spacing is much more important than the austenitic grain size in enhancing the strength. At high strain, twin boundary breaks up for the intensive interaction between it and dislocations.

The mechanical properties obtained in the tensile test strongly depend on the microstructure obtained from the cold rolling deformation process. The HNSS possesses a good balance between strength and ductility in the entire rolling strain range. A detailed report on the results for the effect of cold rolling on other mechanical properties and microstructure of HNSS is beyond the framework of the present paper and will be published separately.

Summary

The mechanical property and microstructure of nitrogen alloyed austenitic steels with different cold rolling strains were systematically investigated. With increasing cold rolling strain from 0 to 70%, the strength of HNSS increased but the ductility declined. Our HNSS exhibits a good balance between high strength (1001–2236 MPa) and satisfactory ductility (12.3–86.6%) compared with classical stainless steels. This is related to the gradual changes of deformation mechanisms derived from the cold rolling deformation. The microstructure investigation revealed that three different deformation mechanisms operated at different rolling stages. As cold rolling strain increased, the dominant mechanisms were changing from coupling operation of early slip and twinning to drossy twin mechanism. In addition, the width of twins decreases with increasing cold rolling strain, which changed from micro- to nanometre range. The cold rolling deformation could be a new way to prepare bulk nanotwin structure materials.

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