Microstructures and deformation behavior of nano-grained and ultrafine-grained high-Mn austenitic steel fabricated by asymmetric-symmetric rolling

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Abstract
The nano-grained (NG) high-Mn austenitic steel with average grain size of 60 nm and the ultrafine-grained (UFG) steels with grain size below 500 nm are successfully produced by combination of the asymmetric rolling (ASR) and symmetric rolling (SR) method and the subsequent annealing treatment. The annealed NG steels exhibit relatively higher strain hardening and good balance of strength and ductility, which is attributed to the partial recrystallization of the nanostructures and the relatively lower stacking fault energy of the high-Mn TWIP steel.

Key Words: Ultra-fine grained (UFG) materials; High-Mn austenitic steel; Asymmetric rolling (ASR); Symmetric rolling (SR);

1. Introduction
During the past two decades, high-Mn austenitic twinning induced plasticity (TWIP) steels with relatively low stacking fault energy (SFE) have received considerably scientific and technological interest, due to their unique comprehensive mechanical property with excellent combination of the high strength and ductility [1-10]. Nevertheless, compared with existing advanced high strength steels (AHSS), high manganese austenitic steels usually show relatively low yield strength (YS), which, to some extend, limits their immediate implementation, particularly for anti-intrusion (crash) assemblies[5]. Grain refinement is proved to be effective.
method to improve the low YS of TWIP steels\cite{2,5,11,12}.

Fabrication of the Nano-grained (NG) and Ultrafine-grained (UFG) materials by severe plastic deformation (SPD) has been attracting a great deal of attention of the materials research community over the past half century. The UFG metals possess much desired high strength but usually disappointingly low ductility compared with their coarse-grained (CG) counterparts due to inability of sustaining the high strain hardening rate to large strains, which severely limits their practical application\cite{13}. Nevertheless, most recent studies show an interesting result that the UFG TWIP steels produced by heavy rolling and subsequent annealing can exhibit good balance of strength and ductility although the TWIP has no longer the dominant deformation mode\cite{2}.

Various SPD processing methods have been attempted to produce the UFG high-Mn steels, e.g. high-pressure torsion (HPT)\cite{14}, equal-channel angular pressing (ECAP)\cite{1,15,16}, accumulative roll-bonding (ABR)\cite{15}, etc. However, the UFG materials prepared by these SPD processes are usually small dimensions, which greatly limit their extensively industrial application.

Rolling is the most suitable way to produce larger sheet or plate type materials. Unlike symmetric rolling (SR), Asymmetric rolling (ASR) involves different circumferential velocities of the two working rolls due to their different diameters or rotation speeds, which greatly enhance the total strain applied to the materials. This method has been proved to possibly satisfy the requirements of an SPD process\cite{17}. In our co-worker’s previous works, ASR has been utilized to successfully produce UFG Fe\cite{18}, Al\cite{19} and Ti\cite{20}. To the best of the author’s knowledge, no information has ever shown that NG and/or UFG high-Mn austenitic steel was produced by ASR method so far. In the present study, the method of cold-ASR combined with SR process is attempted to develop the NG high-Mn austenitic steel. The obtained steels were subsequently annealed at different temperatures and their microstructures, properties and deformation behavior were studied.

2. Experimental

The high-Mn steel with a chemical composition of Fe-25Mn-2.8Al-2.4Si-0.08C (wt.%), which comprises a single phase of austenite at room temperature and is also known as a TWIP steel, was used in the present study. A hot-rolled plate of this steel with a thickness of 20 mm was annealed firstly at 950 °C for 2 h followed by air cooling and then cold asymmetrically rolled (ASR-ed) to a thickness of 2 mm and then cold symmetrically rolled (SR-ed) to a thickness of 0.42 mm with a total reduction of 98%. The rolling was done in multiple passes under lubrication. The cold-rolled sheet was subsequently annealed at 400, 500, 600 and 700 °C for 30 min. Uniaxial tensile tests were performed at ambient temperature using dog-bone-shaped specimens with a
gauge length of 20 mm and a gauge width of 3.5 mm at strain rate of $5 \times 10^{-4} \text{s}^{-1}$. Transmission electron microscopy (TEM) observations were performed using a JEM-2100 microscope operating at 200 kV. TEM specimens were prepared by twin-jet electro-polishing in a solution of 7% HCLO$_4$ and 93% CH$_3$CH$_2$OH at -30 °C.

3. Results and discussion

The optical micrographs of the high-Mn steels for different processes are shown in Fig. 1. The steel by hot-rolled and 950 °C annealed comprises equiaxed grains with an average grain size of about 25 µm and some annealing twins (Fig. 1(a)). After 98% cold ASR-ed and SR-ed, the grains disappear and some very narrow fluid-like shear bands are clearly observed, indicating the severely plastic straining occurs in the material, as shown in Fig. 1(b). During the rolling deformation processing, the nanosized banding structure involving high dislocations density is often formed in high-Mn steels, which is believed to significantly enhance formation of recrystallized grains upon subsequent annealing$^{[2]}$. The microstructure of annealed sample at 500 °C also shows shear banding structure which is similar to the cold rolled steel. However, some dark lines of the banding lamellar have been broken and some finer grain-like area are visible (Fig. 1(c)), indicating the steel is partially recrystallized. With the increasing annealing temperatures, the extent of recrystallization increases. When the sample was annealed at 600 °C, the macro-banding nearly disappear, as presented in Fig. 1(d). Despite the occurrence of the rugged surface in the sample, but it seems that most of the microstructures have fully recrystallized and most of these recrystallized grains are NGs or UFGs. Completely recrystallized microstructures are obtained in the sample annealed at 700 °C, as shown in Fig. 1(e) and it is believed the average grain size for these recrystallized grains are under 500 nm.
A typical TEM microstructure and selected area diffraction (SAED) pattern of the annealed sample at 500 °C are shown in Fig. 2. The SAD pattern is ring shaped, indicating that the nanostructure involves large misorientations. The average size of the steel annealed at 500 °C from the dark field images has been estimated to be 60±12 nm. It was evident that the grain of cold ASR-ed-SR-ed steel and the annealed steel at 400°C was much finer.
Fig. 2 Typical TEM morphologies of the high-Mn steel ASR-ed+SR-ed to 98% reduction in thickness and then annealed at 500 °C for 30min: (a) bright field image and (b) dark field image. The inset shows the corresponding SAED pattern.

XRD patterns of the high-Mn steels are presented in Fig. 3. The diffraction peaks are obviously broadened and the preferred (110) and (111) orientations were considerably increased for the 98% ASR-ed-SR-ed and the annealed steels, in comparison to the hot-rolled and annealed steel. With the increasing annealing temperatures, the relative intensity of the preferred (110) and (111) peaks reduced. Interestingly, the (100) and (311) peak is almost eliminated for ASR-ed-SR-ed steel, but appears again after annealing at 700 °C, which seems indicating the annealed samples at 700 °C is close to the initial state of the cold rolled steel.
Fig. 3 XRD patterns of the high-Mn steel by cold-ASR-SR-ed and annealing at different temperatures.

Fig. 4 displays the engineering stress-strain curves and the corresponding strain hardening rate curves of the testing specimens for different processes. The strength of the high-Mn steel by 98% ASR-ed plus SR-ed and the subsequent annealing is significantly enhanced. Noticeably, the yield strength of nano-structured high-Mn steels annealed below 600 °C exceeds 1000 MPa. Without considering the influence of the composition, this is significantly higher value than the UFG high-Mn steel reported by the mostly recent study\textsuperscript{[2]}. In addition, the annealed UFG austenitic steel at 700 °C shows high strain hardening and a very good mechanical property with high strength and better elongation, which should be contributed to the fully recrystallized nanostructures making the deformation twins to form during tensile deformation. Interestingly, compared with the cold rolled (CR) and 400°C annealed NG steels, the NG high-Mn steels annealed at 500 and 600°C also exhibit higher strain hardening and relatively good balance of strength and ductility. UFG materials produced by SPD usually have high strength but disappointingly low ductility. The low ductility of UFG materials is attributed to the lack of work hardening caused by their inability to accumulate dislocations due to their small grain sizes and saturation of dislocations. It was found that twinning in high-Mn austenitic steel is greatly prohibited when the grain size is reduced to about one micron\textsuperscript{[2, 7]}. TWIP is not the dominant plastic mode for the present high-Mn NG steels. The relatively large elongation of the 500 and 600 °C annealed NG steels should contribute to the dislocations recovery during annealing and partial recrystallization of the nanostructures. As for the higher strain hardening in NG steels, it is presumably due to the lower stacking fault energy, which inhibits dynamic recovery of
dislocations by cross slip and climb and enhances the accumulation of the dislocations during the tensile deformation\cite{13}.

Fig. 4 Tensile engineering curves of the 950 °C annealed hot-rolled (HR), cold-ASR+SR (CR) and various annealing temperatures, Inset: the enlarged tensile curves for CR and 400 °C annealing (a) and the corresponding strain hardening rate (b)

Compared with the annealed steel at 700°C, the annealed steels below 600°C exhibit
relatively low strain hardening although the strength is very high. This is possibly because the nanostructured austenitic steel is partially recrystallized. Essentially, the NG or UFG materials by SPD are naturally deformation microstructures. It was found that bimodal grain size distribution consisting of a mixture of different grains is effective in achieving the best combination of strength-ductility in UFG materials. To retain some part of unrecrystallized structures in high-Mn steels, to some extent, is effective method to obtain and excellent combination of strength and ductility. In comparison of the annealed NG steel at 400 and 500°C, the NG steel annealed at 600°C exhibits relatively larger strain hardening and good balance of strength and ductility. Evidently, controlling the extent of recrystallization is the key factor in achieving the excellent comprehensive mechanical property of the UFG materials.

4. Conclusion

The nano-grained (NG) high-Mn austenitic steel with grain size of 60nm is successfully produced through heavy cold-processing by combination of the asymmetric rolling (ASR) and symmetric rolling (SR). The effect of the annealing temperatures on the microstructures and properties of the NG steels are studied. The results show that the obtained NG and UFG high-Mn steels exhibit relatively higher strain hardening and good balance of high strength and ductility. This is possibly due to the relatively lower stacking fault energy of the steel and the partial recrystallization of the nanostructures.

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