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Enhanced mechanical properties in a high-manganese austenitic steel through formation of nano grains, nanotwinned austenite grains, nano carbides and TRIP

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ABSTRACT

In this work, a specific thermomechanical treatment was used to enhance mechanical properties of a high-manganese austenitic steel. Simple uniaxial tension tests revealed that the treated steel exhibits an outstanding strength (yield strength: 970 MPa and tensile strength: 1384 MPa) and ductility (fracture strain: 37%) synergy. Microstructural studies showed that several strengthening mechanisms contribute to mechanical properties of the treated steel, including nano grains, nano twinned austenite grains, nano carbides and TRIP. Stacking fault energy and strain-induced martensitic transformation were found to have an important role in appropriate adjustment of the strengthening mechanisms to achieve the optimum properties.

1. Introduction

Over the past years, extensive studies have been performed to develop next generation steels with significantly enhanced strength–ductility synergy for light weight structural applications. Of them, high-Mn austenitic steels have received special attention owing to their excellent mechanical properties, which are caused by strain-induced α′-martensitic transformation or mechanical twinning, depending on Mn content. These plasticity mechanisms are termed transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP) [1]. Various strengthening methods have been employed in high-Mn austenitic steels to take full advantages of their unique characteristics and achieve excellent strength–ductility synergies, including nano/ultrafine grains [2–4], nano twinned austenite grains (nt-γ) [5,6], nano precipitates [7] and their combination with TRIP/TWIP [8,9]. In the last case, TRIP/TWIP promotes the ductility and formation of highly dispersed nano precipitates or nano/ultrafine grains increases the strength.

It has been shown that in Fe–Mn alloys with 10–14 wt% Mn, microstructure contains ε-martensite and α′-martensite at room temperature [10]. These alloys mainly reveal TRIP effect during deformation. With increase of Mn concentration to 14–27 wt%, the alloy exhibits only ε-martensite without α′-martensite and the TRIP/TWIP effects coexist [10]. Most of the previous researches on the development of ultrahigh strength high-Mn austenitic steels have been devoted to stable Fe–Mn–C and Fe–Mn–Al–Si grades in which deformation twinning is the governing deformation mechanism. Whereas, metastable austenitic steels are readily prone for effective grain refining through martensite reversion as demonstrated in earlier studies [11–13]. In addition, optimized occurrence of TRIP in these steels improves significantly the strength–ductility synergy [14]. Therefore, it might be possible to utilize the combination of above strengthening mechanisms in a TRIP/TWIP austenitic steel to achieve better mechanical properties than single TWIP or TRIP steels.

In the current work, we present promising mechanical properties obtained in an Fe–4Cr–16Mn–0.1C (wt%) austenitic steel through the application of a specific thermomechanical treatment composed of hot-rolling, cold-rolling and reversion annealing processes. Microscopy techniques and tensile tests are used to explore the microstructural evolutions during thermomechanical treatment and the underlying mechanisms contributing to the mechanical properties of the alloy. It is shown that all of the

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above-mentioned strengthening mechanisms occur in the microstructure of the treated steel.

2. Material and experimental procedure

In this work, raw material of Fe–3.9Cr–15.9Mn–0.09C (wt%) steel was provided in the form of a rod with a diameter of 100 mm. Some blocks of $5 \times 5 \times 100\ mm^3$ were cut from the raw material and then hot-rolled (HR) to a thickness of 2 mm at 1173 K followed by water quenching. These sheets were cold-rolled (CR) to 1 mm thickness (~50% reduction), and finally reversion annealed (RA) at 973 K for 100 s followed by water quenching.

X-ray diffraction (XRD) tests of the samples were performed at room temperature in the range of 40–100 °C with Cu-K_α radiation using a Phillips Expert (MPD) equipment. An optical microscope (OM; Olympus, CX51) was employed to measure the grain size of the samples. Due to the limitation in the resolving power of optical microscopes, a field-emission transmission electron microscope (FE-TEM; JEOL, JEM 2100F) equipped with an energy dispersive X-ray spectrometer (EDXS; Olympus, INCA Energy) was used to characterize the microstructure of the fine grain samples. Specimens for microstructural characterization were first electrolytically polished using solution consisting of 60 ml HNO_3, 180 g CrO, 3 ml HCl, 240 ml H_2SO_4 and 600 ml water at room temperature under 45 V. Then, they were electroetched using a mixed solution of 25 ml HNO_3, 30 g CrO, 3 ml CH_3COOH and 20 ml water under 1.4 V. TEM specimens were prepared by mechanical polishing and subsequently by electropolishing with a twin-jet electro-polisher (Struers, TenuPol-5) in a solution of 90% acetic acid glacial and 10% perchloric acid at 293 K under 35 V. Flat tensile specimens were cut from the annealed sheets using the electron discharge machining (EDM) method, according to the standard technique ASTM E8 and parallel to the rolling direction with the gauge length of 25 mm. Uniaxial tensile tests were carried out three times per each alloy with an Instron-type tensile machine at 298 K and a strain rate of 0.001 s^{-1}. A field-emission scanning electron microscope (FE-SEM; Hitachi, S4160) was used to study the fracture morphologies of the rupture samples. The volume fractions of the α′-martensite in alloys were measured using a Ferritescope (Fisher, M30).

3. Results and discussion

Fig. 1a shows OM micrograph of the raw material. It is observed that the starting material has a dual phase microstructure composed of austenite and ε-martensite. The red arrows in Fig. 1a indicate the occurrence of ε-martensite. The grain size (GS) of the raw material is measured to be 65 μm.

Fig. 1b represents OM micrograph of the HR steel with decreased GS of 17 μm. The needle-shaped morphology of ε-martensite is still discernable in the HR steel. OM micrograph of the 50% cold-rolled steel (CR) is shown in Fig. 1c. In this figure, red and black arrows indicate ε- and α′-martensite, respectively. Ferritescope measurements revealed the formation of 76 vol% deformation-induced α′-martensite in the CR steel. Fig. 1d shows XRD patterns of the raw material, HR and CR steels. Bright field and selected area diffraction pattern (SADP) images of the RA steel can be seen in Fig. 1e and f, respectively. It is observed that the GS is effectively decreased to 150 nm, which is the smallest grain size among those reported for high-manganese austenitic steels (0.35–2.1 μm) [3,4,15]. It can be seen that RA steel has a bimodal microstructure composed of nano ($d < 100$ nm) and ultrafine ($d < 500$ nm) grains of austenite. Also, it is discernable that RA steel has a fully austenitic microstructure without martensite (Fig. 1f).

Previous investigations show that the strain-induced martensite and its reverse transformation (SIMRT) process can be successfully employed to make NG ($d < 100$ nm)/UFG ($d < 500$ nm) austenitic steels [16–25]. The SIMRT process includes cold rolling to form strain-induced α′-martensite from metastable austenite (γ) and annealing for the reversion from martensite to γ, leading to noticeable grain refinement. The strengthening mechanism in the NG/UFG structures is due to the grain refinement strengthening, as known from the Hall–Petch relation [26]. Usually heavy cold rolling (70–90% reduction) is inevitable to achieve nano grain, which limits application of this technique in practice. Low stacking fault energy (SFE) enhances SIM formation and accordingly decreases the amount of required cold rolling for effective grain refinement [27].

SFE of the raw material can be calculated using the basic equation developed by Olson and Cohen [28], as follows:

$$SFE = 2\rho(\Delta G^{\gamma\to\varepsilon} + E^{str}) + 2\sigma(n) \quad (1)$$

where ρ is the density of atoms in a close packed plane in moles per unit area, $\Delta G^{\gamma\to\varepsilon}$ is the chemical free-energy difference between the austenite and ε-martensite phases, $\sigma(n)$ is the surface energy and E^{str} is the strain energy, which in the case of $\gamma\to\varepsilon$ transformation is small. Based on the work of Dumay et al. [29], $\Delta G^{\gamma\to\varepsilon}$ can be calculated as follows:

$$\Delta G^{\gamma\to\varepsilon} = \Delta G^{\gamma\to\varepsilon}_{FeMnCr} + x_c \Delta G^{\gamma\to\varepsilon}_{FeMnCr/C} + \Delta G^{\gamma\to\varepsilon}_{mg} \quad (2)$$

where $\Delta G^{\gamma\to\varepsilon}_{FeMnCr}$ is the chemical contribution of all the elements in substitution in the fcc lattice,$\Delta G^{\gamma\to\varepsilon}_{FeMnCr/C}$ accounts for an increasing carbon effect (as a disturbance of the former fcc solid solution) with manganese content and $\Delta G^{\gamma\to\varepsilon}_{mg}$ is a magnetic term, due to the Néel transition (paramagnetic to antiferromagnetic). By substituting the corresponding values of ρ, $\Delta G^{\gamma\to\varepsilon}$ and $\sigma(n)$, SFE of the raw material at 293 K is calculated to be 2.9 mJ/m^2, implying that the austenite of studied steel is highly metastable owing to its low SFE.

In addition to nano grains (NG), the occurrence of nano precipitates (P) and nano twinned (nt) austenite grains was observed in the RA steel, as shown in Fig. 2a. Fig. 2b shows a high-resolution image of the precipitates, which is determined to be $(Fe,Mn)_3C$ carbides with an average size of 20 nm. The mean spacing of the nano-scale mechanical twin/austenite matrix lamellae is around a few tens of nanometers. Recently, it has been shown that nano twinned austenite (nt-γ) grains possess a yield strength of a few GPa with very high strain-hardening capability and austenitic steels strengthened by these grains exhibit a superior combination of strength and ductility [30].

It has been reported that nano-scale twins are effective in strengthening of austenitic steels without scarifying the plasticity. The enhanced strength–ductility synergy originates from the fact that twin boundaries (TBs) are effective in blocking dislocation motion, and at the same time TBs can act as slip planes to accommodate dislocations [31]. Since twinning in austenitic steels is favored at higher strain rates and/or lower temperatures, plastic deformation at high strain rates, namely dynamic plastic deformation (DPD), is preferable for generating nano scale twins. Therefore, formation of nt-γ grains in the studied steel through the employed thermomechanical treatment can be an alternative approach to take advantages of this method.

Fig. 3 compares engineering stress–strain curves of the raw material, HR and RA steels. The raw material has a yield strength of 394 MPa, tensile strength of 564 MPa and a very small tensile elongation of 3%. The Ferritescope measurements on the fracture surface of the tensile sample showed a fully martensitic microstructure. HR steel possesses higher yield strength of 450 MPa. In contrast to the raw material, tensile strength and elongation considerably increase to higher values of 1131 MPa and 37%,