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Revealing the mechanical properties and microstructure evolutions of Fe–22Mn–0.6C–(x)Al TWIP steels via Al alloying control

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ABSTRACT

Three kinds of Fe–22Mn–0.6C–(x)Al (wt%) Twinning-Induced Plasticity (TWIP) steels were designed by changing the Al content. Uniaxial, unloading-reloading and stress-relaxation tensile tests were carried out to evaluate the mechanical properties. Meanwhile, electron channeling contrast imaging (ECCI), transmission electron microscopy (TEM) and X-ray tomography (XRT) techniques were used to investigate the microstructure evolutions. Firstly, it was found that the Al addition reduced the back stress level, suppressed twinning capability, and increased the stacking fault energy (SFE), leading to concession of work-hardening rates. Secondary, the Al addition improved the friction stress level and promoted the short-rang order (SRO) effect, which brought the transformation of dislocation slip from wavy to planar mode. Last but not least, the dynamic strain aging (DSA) effect brought the pinning of dislocations, forming high dislocation activation volume ($> 100\ b^3$). With increasing plastic strain, the deformation twins and SRO effect restricted dislocations gliding, resulting in low dislocation activation volume ($< 50\ b^3$). The present study could further illustrate the effects of SFE, SRO and DSA on the mechanical properties and microstructure evolutions of Fe–Mn–C–(Al) system TWIP steels via Al addition.

1. Introduction

Twinning-induced plasticity (TWIP) steel has fully austenitic structure with low stacking fault energy (SFE, e.g. 20–60 $mJ\,m^{-2}$) [1–3]. The progressive formed nano-scale deformation twins (DTs) divide the grains into small domains and result in considerably high work-hardening rate [4–6]. Therefore, TWIP steel has outstanding combination of uniform elongation (UE) and ultimate tensile strength (UTS) [7]. In the early year of 1888, Robert Hadfield developed a high-manganese austenitic steel, which had excellent wear resistance and nonmagnetic characteristics [7]. Later, Grassel et al. [8] proposed a high-manganese austenitic steel with Al and Si addition, which exhibited extraordinary shock resistance and deep drawability. Soon after that, Bouaziz and Guelton [9] found that the Fe–27Mn–0.02C (wt%) steel also had TWIP effect. In general, the TWIP steel family has two typical systems: i.e. Fe–Mn–Si–Al and Fe–Mn–C TWIP steels, and the Fe–Mn–C system TWIP steel has better combination of UE and UTS [10]. However, some disadvantages of Fe–Mn–C system TWIP steel need to be solved as below.

1. Negative strain rate sensitivity: the flow stress and UTS of Fe–Mn–C TWIP steel decrease with increasing the strain rate [11–13]. The weakened mechanical properties during high strain rate deformation will restrict the capacity of crash energy absorption. The dynamic strain aging (DSA) effect is considered to be the primary cause [10,14].
2. Hydrogen-induced delayed fracture: after deep drawn cup test, some edge cracks appear in Fe–Mn–C system TWIP steel after a certain time [15,16]. The spontaneous cracking could destroy the product quality of TWIP steels and restrict their industrial applications. This delayed fracture has a strong connection with hydrogen. The accumulation of hydrogen atoms at preferential sites, such as grain and twin boundaries (TBs), increases the residual stress and gives rise to delayed fracture [17].
3. Premature fracture: the Fe–Mn–C TWIP steel exhibits slant fracture without necking, and the work-hardening rate dramatically drops at the fracture strain [18,19]. This slant fracture results from the DSA effect, which promotes abrupt shear fracture along the localized deformation bands (i.e. Portevin-Le Chatelier (PLC) band [20,21]). This unpredictable slant fracture could induce failure without any indications.

To overcome the shortcomings of Fe–Mn–C system TWIP steel, the addition of Al is adopted to mediate the mechanical properties. Firstly, the Al addition suppresses the DSA effect, and then the negative stain

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rate sensitivity can be converted into positive value [10,22]. Secondly, the Al addition transforms the fracture mode from shearing to necking, and avoids the unpredictable premature fracture [22]. Thirdly, α-Al_2O_3 layer forms by Al addition to prevent H permeation [23], so that the hydrogen-related delayed fracture can also be inhibited. Furthermore, the Al addition can reduce the density and enhance the corrosion resistance [24]. Although the Al addition can ameliorate the mechanical performance of Fe–Mn–C TWIP steel, it also exerts negative effects. For instance, the Al addition weakens the deformation twinning capability and work-hardening rate [25]. Besides, the Al addition brings aluminide inclusions [26], which could deteriorate the plasticity.

Above all, effects of Al addition on mechanical properties and microstructure evolutions of Fe–Mn–C system TWIP steels are important but also complex. In the previous studies, the strain rate test reveals the pronounced effect of DSA on strain rate sensitivity, and a DSA-assisted twinning mechanism is proposed [27,28]. The Al solid solution hardening, post-uniform elongation, tensile fracture mechanism, and dynamic Hall-Petch effect are also discussed in Al-added Fe–Mn–C TWIP steels [22,25,29]. In the present study, we apply uniaxial tension, unloading-reloading and stress-relaxation tests, cooperating with advanced microscopy observation methods, to gain a deep understanding on the mechanical properties and microstructure evolutions of Fe–Mn–C system TWIP steels via Al addition.

2. Material and methods

2.1. Materials preparation

Fe–22Mn–0.6C (wt%, denoted as 0Al), Fe–22Mn–0.6C–3Al (wt%, denoted as 3Al), and Fe–22Mn–0.6C–6Al (wt%, denoted as 6Al) TWIP steels were selected in the present study. The preparation of Fe–22Mn–0.6C–(x)Al TWIP steels can be found in a previous paper [10], and their detailed parameters are listed in Table 1.

2.2. Tensile test

Dog-bone tensile samples were spark cut along the axial hot-forged direction, with a gauge dimension of 3 × 3 × 1 mm^3. All the samples were mechanically polished to $2000^{\#}$ SiC paper and electronically polished using a solution of 90% glacial acid and 10% perchloric acid under 12 V. Tensile tests were performed with INSTRON 5982 testing machine at room temperature with an initial strain rate of 10^{-3} s^{-1}.

Uniaxial tension test: the strain was recorded by extensometer up to a true strain of 0.08, and then the crosshead displacement was adopted to record the rest plastic strain. **Unloading-reloading test**: the strain was recorded by an extensometer during the tensile test. At a certain strain, the specimen was unloaded in a displacement-control mode with an unloading rate of − 0.9 mm min^{-1}. **Stress-relaxation test**: the strain was recorded by an extensometer during the test. Specimens were strained up to certain strains, and then the crosshead was fixed and the stress was recorded with time. After stress-relaxation test with time between 20 and 40 s, specimens were reloaded to the previous stress level.

Table 1
Nominal chemical composition, stacking fault energy and density of the selected TWIP steels.

Materials	Composition (wt%)						SFE (mJ m^{-2})	Density (g cm^{-3})
	Mn	C	Al	P	S	Fe		
0Al	21.7	0.66	–	0.007	0.004	Bal.	19	7.83
3Al	20.4	0.59	3.24	0.008	0.005	Bal.	42	7.38
6Al	21.3	0.61	5.81	0.008	0.004	Bal.	66	7.12

2.3. Microstructure observation

As-received samples of 0Al, 3Al and 6Al TWIP steels had fully austenitic structure as shown in Fig. 1(a), and no preferred texture was found by electron backscattered diffraction (EBSD) [10]. Microstructures of 0Al, 3Al and 6Al TWIP steels were also detected under optical microscope (OM) observation, as shown in Fig. 1(a–c). 0Al, 3Al and 6Al TWIP steels all have fully recrystallized microstructure, with annealing twins inside the grains. Average grain sizes of 0Al, 3Al and 6Al TWIP steels without consideration of annealing twins are 40 ± 16 μm, 32 ± 7 μm and 32 ± 6 μm, respectively. When the annealing twins are considered, grains sizes of these steels are measured to be 23 ± 10 μm, 25 ± 8 μm and 27 ± 16 μm, respectively.

Microstructure observations after tensile tests were carried out using a LEO Supra 35 field emission scanning electron microscope (FE-SEM) equipped with electron channeling contrast imaging (ECCI) component under 20 kV, and an FEI Tecnai F20 transmission electron microscopy (TEM), operating at 200 kV. TEM thin foils were spark cut from the tested specimens along the loading direction, and then mechanically polished and perforated under a twin-jet electro-polisher at 36 V with the electrolyte of 90% glacial acetic and 10% perchloric acid at room temperature. Annealed samples after electro-polish were detected by X-ray diffraction (XRD) using a diffractometer with Cu target at a scanning rate of 4° min^{-1} from 40° to 100°. XRD results of fractured TWIP steels are illustrated in Fig. 1(d), in which no secondary phase is found after fracture. Fracture regions of these samples were inspected by a Versa XRM-500 three-dimensional X-ray tomography (3D-XRT) with the volume of around 1.2 × 1.2 × 2 mm^3 and a resolution of 2.5–3 μm per pixel. Voids were marked by different colors depending on the volume size.

3. Experimental results

3.1. Tensile tests

Uniaxial tensile test: true stress-strain curves of 0Al, 3Al and 6Al TWIP steels are illustrated in Fig. 2(a). The ultimate tensile strength (UTS) and uniform elongation (UE) decrease with increasing the Al content. It is worth noting that the flow stress of 6Al steel is higher than that of 0Al and 3Al steels before the strain of 0.3 as inserted in Fig. 2(a). This is because the Al addition raises the yield and flow stress by solid solution hardening [8]. The decrease of UTS and UE with Al addition is strongly attributed to the suppressed work-hardening behavior as compared in Fig. 2(b), which is induced by the increased SFE of Fe–Mn–C system TWIP steel [6,25]. As shown in Fig. 2(c and d), regular serrations are detected in 0Al TWIP steel. However, we did not observe any regular serrations in the entire tensile curves of 3Al and 6Al TWIP steels (Fig. 2(c and d)). These results support the fact that the DSA effect works in 0Al steel but can be highly restrained by Al addition [25,30].

Unloading-reloading test: unloading-reloading curves of 0Al, 3Al and 6Al TWIP steels are illustrated in Fig. 3(a). With increasing the strain, hysteresis loops become larger as shown in Fig. 3(b and c). This transition means that the back stress gets stronger with strain in the three TWIP steels. From the unloading-reloading hysteresis loops, back stress X, and friction stress R can be calculated as below [31]:

$$X = \frac{\sigma_{max} + \sigma_{re}}{2}, \tag{1}$$

$$R = \frac{\sigma_{max} - \sigma_{re}}{2}, \tag{2}$$

where σ_{max} and σ_{re}, as shown in Fig. 3(d), represent the stress just before unloading and the stress departs from linear stress-strain curve, respectively. It should be noted that the measurement of σ_{re} depends on the deviation with a given value, denoted by δ_ε [32]. According to the previous studies [20,32], δ_ε = 2.5 × 10^{-4} is selected in the present study.