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#### Full length article

# Equal-channel angular pressing and annealing of a twinning-induced plasticity steel: Microstructure, texture, and mechanical properties



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#### A R T I C L E I N F O

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#### ABSTRACT

In this work, a high-manganese Fe-23Mn-1.5Al-0.3C Twinning-Induced Plasticity (TWIP) steel was subjected to plastic shear deformation using Equal-Channel Angular Pressing (ECAP) at 300 °C following route B<sub>C</sub> and additional annealing. The microstructure evolution during both deformation by ECAP and subsequent annealing was investigated and correlated with the mechanical properties. The successive grain refinement during ECAP was promoted by two parallel mechanisms, namely dislocation driven grain fragmentation and twin fragmentation, and accounted for the ultra-high strength. In addition, due to the relatively low volume fraction of deformation twins after ECAP at 300 °C, further contribution of deformation twinning during room temperature deformation allowed additional work-hardening capacity and elongation. During subsequent recovery annealing the ultra-fine grains and deformation twins were thermally stable, which supported retainment of the high yield strength along with regained uniform elongation. For the first time, the texture evolution during ECAP and during the following heat treatment was analyzed. After 1, 2, and 4 ECAP passes a transition texture with the characteristic texture components of both high- and low-SFE materials developed. During the following heat treatment the texture evolution proceeded similar to that observed in the same material after cold rolling. Retaining of the ECAP texture components due to oriented nucleation at grain boundaries and triple junctions as well as annealing twinning accounted for the formation of a weak, retained ECAP texture after recrystallization.

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#### 1. Introduction

Twinning-Induced Plasticity (TWIP) steels have gained increased commercial and scientific interest during the past decades. This interest was initiated by the works of Grässel et al. [1,2], who were the first to report the outstanding mechanical properties of these steels. Due to the alloying concepts of these materials, they exhibit a low stacking fault energy (SFE) of ~20 mJ/m<sup>2</sup> to ~50 mJ/m<sup>2</sup> at room temperature [3], which enables the activation of deformation twinning in addition to dislocation slip. As a consequence, high strain hardening rates and thus, high ductility and strength with a typical ultimate tensile strength and elongation to fracture product of more than 50000 MPa % can be achieved [4,5].

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A key factor for achieving the desired mechanical properties of a material is the control and manipulation of the microstructure. With respect to TWIP steels, there have been numerous studies on the influence of precipitation [6,7], deformation [8-14], and recovery and recrystallization annealing [15-23] on microstructure and mechanical properties. Another method to affect the microstructure, which has not yet been studied comprehensively, but is known to have a huge impact on the microstructure, is the application of severe plastic deformation (SPD) to TWIP steels [24-26]. Within the field of SPD methods Equal-Channel Angular Pressing (ECAP), which was developed by Segal and co-workers [27], is the most widely-used and most highly developed technique, due to the relative simplicity of the process and the capability to process large samples [28,29]. Recently, the applicability of ECAP has been extended not only to continuous processing [30,31] but also to consolidation of powders [32-34] and swarf [35]. During deformation by ECAP a high shear strain is imposed into the material as

the specimen passes through the abrupt intersection of entry and exit channel [36,37]. By applying consecutive ECAP passes and varying deformation routes, complex dislocation interactions facilitate a top-down production of bulk ultra-fine grained (UFG) materials having grain sizes in the submicrometer (100–1000 nm) and nanometer (<100 nm) range [38–40]. As a result, ECAP-processed materials reveal strongly enhanced strength, usually combined with good ductility characteristics [38,41].

A material property that is often underestimated but has strong influence on the mechanical behavior, such as plastic anisotropy, formability, and strength, is the crystallographic texture. The previous studies on both experimental description and modeling of texture evolution during ECAP have recently been reviewed by Beyerlein and Tóth [42]. In essence, a simple shear texture is formed during deformation by ECAP due to the deformation mode. However, depending on several factors, such as die geometry, deformation temperature and ECAP route, to name only a few, significant deviations from simple shear textures can be expected.

As mentioned above, the potential of TWIP steels processed by severe plastic deformation has hardly been investigated so far and thus, also the texture evolution has not been analyzed. Recently the influence of deformation temperature and number of ECAP passes on the microstructure and mechanical properties of an Fe-22Mn-0.6C TWIP steel was investigated [43]. In the present study, the deformation temperature was kept constant at 300 °C, whereas the number of ECAP passes was increased up to 4 following route B<sub>C</sub>. The microstructure evolution was characterized comprehensively after each pass and correlated with the texture evolution and the mechanical properties. As mentioned above, the outstanding mechanical properties of TWIP steels originate from their low SFE at room temperature. Due to the deformation at 300 °C the SFE was increased. Especially the material behavior with respect to this change in SFE was investigated, as there are only few studies on the influence of SFE on both microstructure [44-47] and texture development during ECAP [48]. Further, the ECAPprocessed TWIP steel samples were subjected to additional annealing in order to characterize the thermal stability of the deformed microstructure and the recrystallization behavior. Here, the focus was put on the correlation between microstructure and texture evolution.

#### 2. Experimental

#### 2.1. Material chemistry and processing

The exact chemical composition of the investigated TWIP steel is given in Table 1. The SFE at room temperature and at 300 °C was calculated to be ~25 mJ/m<sup>2</sup> and ~75 mJ/m<sup>2</sup> using a subregular solution thermodynamic model [49].

The melt was cast into 100 kg ingots, followed by homogenization annealing of the slabs at 1150 °C for 5 h in a muffle furnace. Then, the 140 mm thick ingots were forged at 1150 °C to a reduced thickness of 55 mm and again homogenized. Afterward, the slabs were hot rolled at 1150 °C to a final height of 10 mm. Rods with a diameter of 10 mm and a length of 35 mm were cut out of the hotrolled slabs perpendicular to the rolling direction for further deformation by ECAP.

The ECAP rig used for experiments is described in detail in Ref.

Table 1   Chemical composition of the investigated alloy.							
Element	Fe	С	Mn	Al	Si	Ν	Р
[wt.%]	Bal.	0.325	22.46	1.21	0.041	0.015	0.01

[50]. In the study of a TWIP steel similar to that investigated in the current work [43], it was found that ECAP of TWIP steel at temperatures lower than 300 °C causes fracture of the punch due to high work hardening of the material. Therefore, the die of the equipment was first heated up to the required temperature of 300 °C and then the specimens were given a time of 5 min to reach the desired temperature in the interior of the die. The TWIP steel rods were pushed from the entry channel through the 90° angle into the exit channel with a feed rate of 1 mm/s, causing shear plastic deformation to occur along the channel intersection plane. ECAP passes of 1, 2, and 4 were realized with air cooling after each pass and a sample rotation of 90° between consecutive passes to follow route  $B_{C}$  [51]. The equivalent strain  $\varepsilon_{N}$  (N is the number of ECAP passes) introduced by ECAP was calculated following the equation in Ref. [38] and resulted in values of  $\varepsilon_1 = 1.155$  after 1 ECAP pass,  $\varepsilon_2 = 2.31$  after 2 ECAP passes, and  $\varepsilon_4 = 4.62$  after 4 ECAP passes. Additional heat treatment was performed in a sandbath furnace at 600 °C.

#### 2.2. Specimens and characterization techniques

Specimens with the dimensions 8 mm  $\times$  10 mm  $\times$  1 mm (extrusion direction (ED)  $\times$  normal direction (ND)  $\times$  transverse direction (TD), respectively) were cut from the deformed rods. The corresponding coordinate system with respect to the die geometry is given in Fig. 1. Sample preparation on the ED-ND sections consisted of mechanical grinding and polishing, followed by electropolishing at room temperature and 22 V using an electrolyte containing 700 ml ethanol ( $C_2H_5OH$ ), 100 ml butyl glycol ( $C_6H_{14}O_2$ ), and 78 ml perchloric acid (60%) (HClO<sub>4</sub>). Transmission electron microscopy (TEM) samples (~100 µm thick, 3 mm in diameter) were prepared using the same electrolyte in a double jet Tenupol-5 electrolytic polisher with a voltage of 25 V at room temperature. In order to reveal the microstructure by optical microscopy, the samples were additionally etched at room temperature using a solution consisting of 2 g potassium disulfide (K<sub>2</sub>S<sub>2</sub>O<sub>5</sub>) and 100 ml cold-saturated Klemm I solution ( $Na_2S_2O_3 + 5H_2O$ ).

Electron backscatter diffraction (EBSD) analyses were performed in a LEO 1530 field emission gun scanning electron microscope (FEG-SEM) operated at 20 kV accelerating voltage and a working distance of 10 mm. EBSD mappings were generated using a step size of 0.15  $\mu$ m and were post-processed utilizing the HKL

Fig. 1. Schematic diagram of the reference coordinate system and sample taking according to the die geometry.



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