Acta Materialia 141 (2017) 374–387

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Designing duplex, ultrafine-grained Fe-Mn-Al-C steels by tuning phase transformation and recrystallization kinetics

Jiali Zhang [a], Dierk Raabe [a], Cemal Cem Tasan [b, *]

[a] Max-Planck-Institut für Eisenforschung, Max-Planck-Straße 1, 40237, Düsseldorf, Germany
[b] Department of Materials Science and Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA, 02139, USA

ARTICLE INFO

Article history:
Received 2 June 2017
Received in revised form
12 September 2017
Accepted 13 September 2017
Available online 13 September 2017

Keywords:
Lightweight steels
Partial recrystallization
Ultrafine grain
Ferrite
κ−carbides
Strain partitioning

ABSTRACT

A novel, lightweight Fe-25.7Mn-10.6Al-1.2C (wt.%) steel is designed by exploiting the concurrent progress of primary recrystallization and phase transformation, in order to produce an ultrafine-grained, duplex microstructure. The microstructure consists of recrystallized austenite grains surrounded by submicron-sized ferrite grains, and recovered austenite regions with preferential nano-κ-carbide precipitation. This partially recrystallized duplex microstructure demonstrates excellent strength-ductility combinations, e.g. a yield strength of 1251 MPa, an ultimate tensile strength of 1387 MPa, and a total elongation of 43%, arising from the composite response by virtue of diverging constituent strength and strain hardening behaviors.

1. Introduction

Driven by the increasing demand for high-strength and energy-efficient metallic materials, high-Mn (20–30 wt.%) Fe-Mn-Al-C steels have drawn significant interest in recent decades. This is due to their superior mechanical properties [1–13] and reduced weight as a result of Al alloying (1.5% density reduction per 1 wt.% Al addition [2,3]). These steels develop primarily two types of microstructures depending on the relative amount of the alloying elements: single-phase austenitic [4,10,12] and duplex[1] ferritic-austenitic [7,8,11]. On the one hand, the austenitic steels are generally reported to have better strength-ductility combinations [1,5,10,12]. On the other hand, the duplex alloys provide a larger variety of possible microstructure variations, although they exhibit inferior mechanical properties attributed to the presence of ferrite (α) [7,9].

In addition to weight reduction, this alloy system attracts interest also due to the strengthening effect caused by a third microstructural constituent, the intragranular nano-scale κ-carbide (ordered $L'1_2$ structure of $(Fe,Mn)_3AlC$) [1,2,4,12,14,15], which can be formed during quenching [4,14,16] or aging treatment [17,18] depending on composition. However, long term aging may lead to the formation of brittle grain boundary (GB)-κ-carbides [19–21], β-Mn [22–24], and for duplex microstructures also B2 [(Fe, Mn)Al] and $D0_3$ [$(Fe,Mn)_3Al$]) in ferrite grains [7,21,25,26]. In addition to the brittle phases, high-Mn Fe-Mn-Al-C steels also suffer from the softening response induced by large grain sizes developed during high temperature homogenization (≥1100 °C) and intensive aging treatments, due to the Hall-Petch effect [27,28]. In fact, thermal treatment optimization to (i) avoid GB-κ-carbide embrittlement [4,17,18,29] and (ii) balance the κ-carbide hardening vs. grain growth softening effects, remains a challenge for these steels.

We solve this challenge and improve the overall strength-ductility combinations of the Fe-Mn-Al-C steels by designing a new microstructure to incorporate strengthening effects of grain refinement and nano-precipitates, while avoiding GB-κ-carbide embrittlement. Inspired by the achieved strength-ductility synergy of a Fe-Mn steel which is partially strengthened by nano-twins [30], we designed a Fe-Mn-Al-C steel microstructure which includes several constituents containing refined austenite (γ) grains or nano-κ-carbides. These microstructural constituents are introduced to contribute to the overall strength-ductility combinations at different stages of deformation, in a composite-like response.

* Corresponding author.
E-mail address: tasan@mit.edu (C.C. Tasan).

[1] This microstructure is sometimes referred to as triplex considering κ-carbides as a third type of phase constituents [2].

https://doi.org/10.1016/j.actamat.2017.09.026
1359-6454/

To realize this microstructure, we developed a thermo-mechanical processing strategy that tunes both phase transformation and recrystallization kinetics by utilizing elemental segregation during diffusional phase transformation constrained recrystallization, i.e. transformation-constrained-recrystallization. The proposed strategy is composed of three steps: (i) cold rolling to enhance the stored dislocation energy and to introduce nucleation sites for recrystallization[2]; (ii) annealing to partially recover and partially recrystallize austenite grains (the latter to an ultrafine-grained scale (UFG, between 1 and 2 μm in diameter [31]), constrained by simultaneous ferrite formation); (iii) quenching for heterogeneous precipitation of κ-carbides (the precipitates are formed already during quenching). In what follows, a full description of this processing strategy as well as the resulting mechanical properties and deformation mechanisms of the obtained partially recrystallized UFG duplex microstructure is given, in conjunction with the discussion of the corresponding reference materials, i.e. the fully recrystallized UFG austenitic and duplex microstructures.

2. Materials and methods

The Fe-25.7Mn-10.6Al-1.16C (wt.%) alloy used in this study was prepared by induction melting under argon atmosphere and cast into a rectangular copper mold. After homogenization in air at 1150 °C for 2 h, the ingot was hot rolled at a starting temperature of 1050 °C from 60 mm down to a hot strip of 2.5 mm in thickness. It was then solution treated (ST) at 1050 °C for 25 min and subsequently water quenched to form a fully austenitic structure. After solution treatment, no macro-segregation was observed. The average grain size (d_γ) was measured to be 20.70 ± 11.01 μm from representative electron backscatter diffraction (EBSD) measurements. The stacking fault energy (SFE) of this steel was reported to be 110 mJ m^{-2} [2]. The as-ST material was cut into rectangular blocks and cold rolled (CR) at room temperature (RT) to 30% ($\varepsilon \approx 0.29$), 60% ($\varepsilon \approx 0.95$), and 90% ($\varepsilon \approx 2.41$) thickness reductions, which are denoted by 30CR, 60CR and 90CR, respectively, hereafter.

In order to accurately control the temperature on the sample, the heating/cooling rate, and the chamber atmosphere, all heat treatments in this study were carried out in a DIL805A/D dilatometer (Bähr Thermoanalyse GmbH) [32]. Temperatures were recorded by a thermocouple welded onto the sample surface. Vacuum and Helium were used respectively as the protecting atmosphere and the cooling agent for heat treatments. Heating rate was 50 K s^{-1}, and an average cooling rate of −220 K s^{-1} was achieved by flushing RT-helium gas at the maximum flowrate. Heat treatments were first carried out at selected temperatures from 300 °C to 900 °C with 5 min durations on the 60CR and 90CR materials to study phase constituents. Recrystallization treatments were then performed on the 60CR material. Tensile samples were heat treated individually. The temperature gradient on a tensile sample was measured with three thermocouples on the head, shoulder, and gauge center to ensure gauge section temperature homogeneity within 5 °C.

All samples probed by scanning electron microscopy (SEM) were wet-ground and polished. Final polishing was carried out using a colloidal silica solution. The microstructure was characterized by employing backscattered electron (BSE) imaging, electron channeling contrast imaging (ECCI) [33], energy-dispersive X-ray spectroscopy (EDS) and EBSD. A JEOL JSM 6500F FEG-SEM (JEOL GmbH) equipped with an EDAX/TSL system (EDAX/TSL, Draper) was used for EBSD measurements. BSE imaging, ECCI, and EDS (at 10 kV) were conducted in a Zeiss Merlin (Carl Zeiss SMT AG) with a Bruker system (Bruker Nano GmbH). The recrystallization fraction was evaluated using two methods: (i) large field EBSD grain average misorientation maps of ~200 × 500 μm^2 in each state; (ii) Argus images (Bruker system), i.e. color orientation contrast images which assign certain color with respect to the crystal orientation by capturing Kikuchi bands [34], with ~1.3 × 1.7 mm^2 area in each state for adequate statistics. All transmission electron microscopy (TEM) samples were prepared with the site-specific method [35] using a dual-beam focused ion beam (FIB) FEI Helios Nanolab 600i. TEM observations were performed in a JEOL JEM-2200 FS (JEOL GmbH) operating at 200 kV, through which bright field (BF), dark field (DF) images and selected area diffraction patterns (SADP) were recorded by a Gatan CCD camera. Scanning transmission electron microscopy (STEM) images were captured by a scanning transmission electron imaging-BF detector with 100 cm camera length. X-ray diffraction (XRD) measurements were conducted using Co-K_α radiation. For easier comparison with literature data, the 2θ values were calculated into the values of Cu-K_α radiation, using wavelengths of 1.54056 × 10^{-10} m and 1.78897 × 10^{-10} m for Cu-$K_{\alpha1}$ and Co-$K_{\alpha1}$. Two methods were used to estimate the ferrite phase fraction: (i) XRD spectra using the method reported by Jatczak et al. [36]; (ii) EBSD datasets with sufficiently large areas.

Dog-bone-shaped tensile samples (gauge geometry: 4 × 2 × 1 mm^3) were machined by electrical discharge machining (EDM) with the gauge length parallel to the rolling direction (RD). Three tensile samples were tested for each condition in a 5 kN Kammrath & Weiss tensile stage with the in-situ imaging using a high speed camera. The data were used for digital image correlation (DIC) analysis by employing the ARAMIS software (GOM GmbH). Microhardness was measured using a Fischerscope® HM2000 micro-indenter equipped with a Vickers diamond indenter. To yield sufficient statistics, indents were mapped as a 10 × 5 pattern on each specimen with a 150 μm spacing and a maximum load of 1000 mN. To study the mechanical response of the microstructural constituents, nanoindentation tests were conducted using a Hysitron Tribolab nanoindentation system with a Berkovich shaped indenter. A trapezoidal load function in a load-controlled mode was used with a constant loading rate of 240 $\mu N \cdot s^{-1}$ and a holding time of 2 s at the maximum load of 1200 μN. A matrix of 10 × 10 indents with a spacing of 10 μm was placed in the center of the recrystallized sample.

3. Results

3.1. Transformation-constrained-recrystallization treatment design

The starting microstructure prior to the partial recrystallization treatment is cold worked. A preliminary study on the 30CR, 60CR and 90CR states reveals deformation by planar dislocation slip and shear banding with neither mechanically induced twinning nor martensitic transformation. The main features characterizing the 30CR and 60CR microstructures are microbands and Taylor lattices, which are clear indications of planar dislocation slip [37,38]. This observation, despite the high SFE of the studied steel (110 mJ m^{-2} [2]), is consistent with previous reports on the deformation mechanisms of similar alloy compositions [5,18], and is likely to be linked with the presence of intragranular κ-carbides. Shear bands prevail in the 90CR microstructure (especially near the roller contact surfaces) and lead to the transition of the main texture component from copper (60CR state) to brass [39–41].

The optimal recrystallization temperature range that would lead to the targeted microstructure was determined by probing the respective phase constituents of the studied alloy system annealed at particular temperatures. This was conducted by analyzing the

[2] The term “recrystallization” is generally referred to as primary recrystallization in this study, if not otherwise specified.