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Nanolaminate transformation-induced plasticity–twinning-induced plasticity steel with dynamic strain partitioning and enhanced damage resistance

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Abstract—Conventional martensitic steels have limited ductility due to insufficient microstructural strain-hardening and damage resistance mechanisms. It was recently demonstrated that the ductility and toughness of martensitic steels can be improved without sacrificing the strength, via partial reversion of the martensite back to austenite. These improvements were attributed to the presence of the transformation-induced plasticity (TRIP) effect of the austenite phase, and the precipitation hardening (maraging) effect in the martensitic matrix. However, a full micromechanical understanding of this ductilizing effect requires a systematic investigation of the interplay between the two phases, with regards to the underlying deformation and damage micromechanisms. For this purpose, in this work, a Fe–9Mn–3Ni–1.4Al–0.01C (mass%) medium-Mn TRIP maraging steel is produced and heat-treated under different reversion conditions to introduce well-controlled variations in the austenite–martensite nanolaminate microstructure. Uniaxial tension and impact tests are carried out and the microstructure is characterized using scanning and transmission electron microscopy based techniques and post mortem synchrotron X-ray diffraction analysis. The results reveal that (i) the strain partitioning between austenite and martensite is governed by a highly dynamical interplay of dislocation slip, deformation-induced phase transformation (i.e. causing the TRIP effect) and mechanical twinning (i.e. causing the twinning-induced plasticity effect); and (ii) the nanolaminate microstructure morphology leads to enhanced damage resistance. The presence of both effects results in enhanced strain-hardening capacity and damage resistance, and hence the enhanced ductility.

Keywords: Reverted austenite; TRIP-maraging; Strain partitioning; TWIP; Damage

1. Introduction

Martensitic steels exhibit high strength but low ductility due to lack of effective hardening and microcrack-arresting mechanisms [1–3]. It has thus been a permanent challenge to improve the ductility of martensitic steels, without sacrificing strength. Different approaches have been explored, focusing on prior austenite state in terms of chemical composition [4–6], grain size [7,8]; defect density [9,10]; tempering of martensite [11,12]; martensite variant selection [13–16]; martensite refinement [4,17,18]; multilayering [19–22], etc. For the same purpose, we have recently introduced a novel alloy and microstructure design concept which we refer to as transformation-induced plasticity (TRIP)-maraging steels [23,24]. In this concept the microstructure is designed in two ways in a single aging treatment step: reverted nanoscale austenite films (γ_{RN}) are formed on the martensite interfaces and nanoparticles are precipitated in the martensitic matrix [25]. In this way these steels benefit from three effects simultaneously, i.e. the TRIP effect [26–30] (from the former), the maraging effect (through the latter) [31], and the classical composite effect. A simultaneous increase in both ductility and strength has already been demonstrated, providing indirect verification of the successful introduction of effective hardening and microcrack-arresting mechanisms in the martensitic matrix through this concept [23,25]. The exact nature of the aging-induced improvements is, however, not fully understood, especially considering the complexity of microstructure optimization required. In this regard, improper chemical composition or thermomechanical treatment considerations may lead to the introduction of additional damage mechanisms (e.g. due to the strain incompatibility of hard (martensite) and soft (γ_{RN}) phases introduced) or ineffective TRIP contribution (e.g. spent early in deformation and absent when needed for damage resistance at high-strain levels).

Therefore in this work, in an effort to provide generalized guidelines for martensitic steel design, we aim to obtain a thorough understanding of the micromechanical processes governing TRIP-maraging steel behavior, i.e. martensite and γ_{RN} plasticity, deformation-induced γ_{RN} transformation and the nucleation and microstructure interaction of damage incidents. For this purpose, employing advanced experimental multiprobe and in situ techniques, (i) the microstructure development in TRIP-maraging steels during austenite reversion process, (ii) the resulting mechanical properties and (iii) microstructural deformation

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and damage mechanisms are systematically studied. In what follows, we first explain this employed experimental multiprobe methodology. Then the microstructure evolution during martensite-to-austenite reversion and its influence on the overall mechanical properties are analyzed. Next, the deformation, damage and failure micromechanisms responsible for the observed mechanical trends are studied in detail, through a variety of both in situ and post mortem measurements. Finally the observations are discussed in comparison to other advanced high strength steels with multiphase microstructures.

2. Experimental procedure

The study was carried out on a TRIP-maraging steel with nominal chemical composition Fe–9Mn–3Ni–1.4Al–0.01C (mass%). Mn thermodynamically stabilizes the austenite, hence enabling martensite-to-austenite reversion upon modest annealing [32,33]. This effect can be used to tune the TRIP effect during the following mechanical test [26–30,34–36]. Al is added to form nanoprecipitates together with Ni and Mn inside the martensite matrix phase, creating the maraging effect [37,38]. We reduced the C content (~0.01 mass%) in order to ensure good weldability and better ductility of the as-quenched martensite [39].

The steel ingot was cast in a vacuum induction furnace and hot rolled at 1100 °C. After homogenization at 1100 °C for 1 h and quenching to room temperature, reversion of α' martensite to austenite was achieved by further annealing at 450 °C or 600 °C for various periods (1 h, 4 h or 8 h) and subsequent quenching in water [25,37,40]. Here we focus on results from as-quenched and 600 °C 1 h and 8 h aged samples (from hereon referred to as S_{aq}, S_{1h}, S_{8h}), which allow us to study the micromechanical details associated with the interaction of dislocation slip, deformation-induced phase transformation and twinning during deformation.

To investigate the influence of reverted austenite (γ_{RN}) on the mechanical properties under both quasistatic and dynamic loading conditions two kinds of mechanical tests were performed. Uniaxial tensile tests were carried out at room temperature with an initial strain rate of $10^{-3}\ s^{-1}$. Impact tests covered a temperature range of −150 °C to 100 °C. For the tensile tests, a Kammrath and Weiss stage was used, and the strain was measured by digital image correlation (DIC) using Aramis software (GOM GmbH). The impact test was carried out on subsize Charpy V-notched samples with a ligament size of $3 \times 4\ mm^2$ machined along the rolling direction according to the standard DIN 50 115. The active deformation mechanisms in the microstructure were investigated by post mortem microstructure characterization of the fractured tensile samples at known strain levels determined by DIC. For these analyses scanning electron microscopy (SEM) based electron backscatter diffraction (EBSD), electron channeling contrast imaging (ECCI) and secondary electron (SE) imaging methods, as well as transmission electron microscopy (TEM) and synchrotron X-ray diffraction (SXRD) analyses were employed. Moreover, in situ SEM tensile and three-point bending tests were performed. The former focused on the analysis of γ_{RN} stability and microcrack-arresting mechanisms. The latter provided insight on strain partitioning between the martensite and γ_{RN} grains.

For the SEM analysis JEOL JSM-6500F (for EBSD, SE), Zeiss-Crossbeam XB 1540 FIB-SEM (for in situ ECCI and in situ EBSD) and Zeiss-Merlin (for post mortem ECCI) instruments were used. EBSD measurements were carried out under an acceleration voltage of 15 kV with a step size of 80 nm. TEM observations were performed in a JEOL JEM-2200FS operated at 200 kV. Specimens for TEM analysis were prepared from 3 mm disks of tensile samples. The sample preparation methodology for SEM and TEM analyses are explained elsewhere [41].

Synchrotron measurements were performed at the high resolution powder diffraction beamline P02.1 at PETRA III (DESY Hamburg, Germany), using synchrotron radiation at a wavelength of $\lambda = 0.20727$ Å. An incident beam with dimension of $200 \times 200\ \mu m^2$ was used. After penetrating through a 1 mm thick sample with known pre-strain values, the two-dimensional XRD patterns were collected using a fast image plate detector Perkin Elmer 1621 (2048×2048 pixels, $200 \times 200\ \mu m^2$ pixel size, intensity resolution of 16 bit), which was placed at a distance of 800 mm to the sample. XRD patterns were integrated into the 2θ space using the software FIT2D [42].

3. Results

3.1. Influence of aging on the microstructure

The partial transformation of α' martensite back to γ_{RN} during annealing at 600 °C is shown in Fig. 1 for the as-quenched sample (S_{aq}), the 1 h heat-treated sample (S_{1h}) and the 8 h heat-treated sample (S_{8h}) as imaged by SE imaging, EBSD phase and inverse pole figure (IPF) maps. In the quenched state, the microstructure is composed fully of α' martensite, as shown by SE imaging of the etched sample surface (Fig. 1a_1) and the EBSD phase map (Fig. 1a_2). No retained austenite is observed within the resolution limit of the high resolution EBSD measurements carried out at a step size of 80 nm (Fig. 1a_2). The EBSD IPF map reveals that the α' martensite laths within one block share nearly the same crystallographic orientation (within 1°) (Fig. 1a_3).

After annealing at 600 °C for 1 h, a new phase with thin layered morphology is formed inside the α' martensite matrix (Fig. 1b_1) and it is indexed as austenite (γ_{RN}) by EBSD (Fig. 1b_2). All the γ_{RN} grains inside one α' martensite block again have similar crystallographic orientations (within 1°) (Fig. 1b_3).

With increasing the annealing time to 8 h, γ_{RN} grains further grow into the α' martensite (Fig. 1c_1–c_3), resulting in the formation of a duplex nanolayered α' martensite–austenite microstructure. In addition, a small amount of ε martensite, i.e. less than 4 vol.%, is also present in the microstructure after the reversion treatment.

The α' martensite and γ_{RN} phases in the S_{8h} specimen (600 °C, 8 h) are revealed in terms of a low magnification EBSD phase map, and further characterized by TEM in Fig. 2. The STEM image (Fig. 2b) provides a higher resolution image of the duplex structure with γ_{RN} grains finely distributed in the α' martensite matrix. Details of the α' martensite and γ_{RN} are revealed by high magnification images in Fig. 2c and d, as guided by the red and green rectangles, respectively. The α' martensite matrix contains nanoprecipitates and a high density of dislocations (Fig. 2c). The γ_{RN} grains, in contrast, are free of