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Micro-damage initiation in ferrite-martensite DP microstructures: A statistical characterization of crystallographic and chemical parameters

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ABSTRACT

Damage mechanisms occurring in a DP-steel microstructure at low strains, under monotonic and cyclic deformation modes have been studied by a comprehensive statistical analysis of scanning electron microscopy observations. The study aims to define local microstructural configurations of high damage susceptibility. In particular, the role of grain and interphase boundaries at triggering micro-damage features was investigated. SEM-based electron backscatter diffraction (EBSD) and electron channeling contrast imaging (ECCI) were utilized. Focused ion beam (FIB) milling was additionally applied for a 3-dimensional investigation of the crack morphology and corresponding arrangement of phases. Microstructural configurations of high damage susceptibility were deduced and confirmed through a quasi in-situ deformation experiment. Damage initiation is shown to be highly dependent on the distribution and morphology of the martensitic islands. Voids are most likely to nucleate at interphase boundaries under monotonic loading, particularly at triple junctions between ferritic grain boundaries and martensite. On the other hand, decohesion cracks are frequently observed inside of martensite where they are remarkably restricted to prior austenite grain boundaries (PAGbs). The low fracture strength of martensitic PAGbs is discussed in the light of local chemical analysis of different types of interfaces using atom probe tomography (APT).

1. Introduction

DP steels comprise one of the advanced high strength steel (AHSS) grades, consisting of hard martensite islands embedded in a soft ferritic matrix. Deformation of a DP-steel microstructure leads to characteristic heterogeneity of the strain distribution, triggered by the mechanical phase contrast at the early stages of deformation [1–10]. This heterogeneity increases with increasing global deformation level, promoting hot spots of strain localization, where damage is most likely to initiate [3,5,9,11,12]. Eventually, DP-steels fail in a ductile manner, following nucleation, growth, and coalescence of the damage features that develop progressively with increasing strain [13].

Different parameters may influence the strain distribution within the DP-steel microstructure, particularly, the average size of the martensite islands and their global distribution within the ferritic matrix [14,15]. More detailed studies investigated the influence of the local distribution and morphology of both phases on the micro-strain localization, identifying strain peaks at critical microstructural configurations [1,7,9,11]. Different mechanisms have been reported for damage initiation, including martensite cracking, damage at interphase boundaries, and void formation inside of ferrite. Though many damage-related studies were concerned with the influence of varying DP-steel parameters on the relative contribution of each mechanism, only few have attempted to define descriptive models for the damage initiation mechanism [16].

Most of the available systematic investigations on DP-steel damage do not provide the relevant crystallographic orientation dependency. On the other hand, in-situ micro-mechanical studies utilizing electron backscatter diffraction (EBSD) often lack the statistical relevance and are limited in terms of the analyzed strain levels due to surface topography effects. Therefore, even though an enormous library of relevant micro-mechanical studies exists, more efforts are still required to attain a microstructural model for damage evolution in DP steels [17] taking into account, at best, the 3-dimensional character of morphological, crystallographic, and chemical factors.

The present study aims to identify more comprehensively independent microstructural features underlying both strain localization and damage. To this end, details of damage initiation mechanisms under monotonic loading in the DP steel microstructure were observed using advanced SEM and FIB observation techniques. In order to

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identify grain and interphase boundaries of low fracture strength, micro-crack propagation was additionally characterized after cyclic deformation. Furthermore, elemental segregation at selected interfaces was assessed using atom probe tomography (APT) to evaluate the chemical aspect of grain or phase boundary embrittlement. In order to observe damage initiation at martensite interfaces already at low levels of deformation, an optimized model DP-steel was utilized with a relatively coarse microstructure and sufficient mechanical phase contrast.

2. Experimental

2.1. Material

Hot-rolled plain C-Mn steel was utilized, with a chemical composition of Fe-0.14C-1.93Mn-0.42Cr-0.25Si (in wt%), and an initial bainitic-martensitic phase composition. A heat treatment process was applied to the specimens under vacuum, using a dilatometer DIL 805 A/D (TA Instruments). The applied process consisted of two isothermal holding steps, first at 840 °C for 90 s for complete austenitization, followed by partial ferritic transformation for 4 min at 650 °C. The material was finally quenched to room temperature. Helium gas was applied for intermediate cooling and final quenching. The final microstructure consisted of ferrite (F) with an average grain size of 3.5 μm, and coarse-lath martensite (M), at an average F:M volume ratio of 7:3.

2.2. SEM characterization techniques

SEM-based techniques are most suitable for crack nucleation studies as they allow – in contrast to TEM – observation of large representative areas within bulk specimens, returning crystallographic and morphological information with sufficient spatial resolution. An ideal technique would allow direct observation of crack initiation in conjunction with crystallographic and strain measurements. This is possible, in principle, by in-situ straining experiments combined with EBSD and digital image correlation (DIC) [5,7,9,18,19]. However, this approach is not without problems since SEM observations are always made very close to the free surface of a specimen where stress-strain distributions differ significantly from the interior. The damage behavior may, therefore, also deviate significantly from that in the bulk specimen. The results also lack information about how the microstructure continues below the surface which renders the interpretation of results highly uncertain as it has been shown, for example, by Diehl et al. [20]. *In-situ* analyses are further restricted by the quick roughening of the surface during straining, particularly for multi-phase materials of high mechanical phase contrast. This spoils precise measurements already at relatively low strain.

As a consequence, here measurements were mostly performed on specimens that were fully metallographically prepared after deformation. The largest disadvantage of this approach is that the local strain remains unknown and can only be estimated from microstructural and crystallographic features. Four different observation modes are available in SEM to obtain strain information, of which three are EBSD-based: the kernel average misorientation (KAM), calculation of grain reference orientation deviation (GROD), and the image quality (IQ). The fourth is the electron channeling contrast imaging (ECCI) technique. These four techniques are illustrated and compared in Fig. 1 by means of an undeformed DP-steel microstructure. The KAM value (Fig. 1c) indicates the average misorientation between each pixel and all its neighbors in a kernel. The GROD value (Fig. 1d) in contrast displays the misorientation of every pixel with respect to a reference pixel in the same grain. For long-range orientation gradients (e.g. in ferrite), GROD values are larger and show grain rotations more sensitively than KAM values. For short range-gradients (e.g. in small martensite lamellae), the KAM value is more indicative. Both values can be interpreted in terms of geometrically necessary dislocation (GND) densities. GNDs are dislocations created as a compensation of different strain rates at different locations in a grain and render a non-zero net Burgers vector. Their density may be assumed to increase with total strain, although there is certainly no linear relationship between both values.

EBSD-based diffraction pattern quality, also named image quality (IQ), may also indicate strain. The IQ value (Fig. 1b) is a measure of the sharpness of EBSD patterns. The lower is this value the higher is the total amount of stored dislocations. This difference is also used to discriminate between martensite (M) with a high defect density and ferrite (F) with a low one. In a pixel-base IQ map grain boundaries, which also have low IQ values, would be falsely assigned to martensite. Therefore, we used the grain-average of the IQ value on which grain boundaries only have marginal effect. Grains with an average IQ value lower than a given threshold are assigned to martensite [17].

In several figures (e.g. Fig. 4) we also use inverse pole figure (IPF) coloring to display orientation maps. These figures indicate the crystallographic direction of one fixed sample direction displayed in a standard orientation triangle overlaid with a color triangle. The displayed sample direction is mentioned in the respective figure captions.

ECCI is an additional SEM technique for direct visualization of dislocations and other extended lattice defects [21]. It is an electron diffraction-based technique similar to bright-field imaging with scanning transmission electron microscopy (STEM). Proper ECCI contrast requires the crystal of interest to be tilted into the so-called two-beam condition, where only one set of lattice planes is tilted into diffraction conditions (Fig. 1a detail **2BC**). Under these conditions individual dislocations and other crystal defects are visible as bright features on a dark background. When the dislocation density increases above ~ 10^{14} m^{-2} the contrast of the dislocations overlaps and no individual dislocations are visible any longer. Instead, these areas show an almost constantly bright backscattering signal. This is the case, for example, for martensite in a DP steel (Fig. 1a detail **M**).

Finally, secondary electron (SE) images allow, due to their high spatial resolution, direct and accurate observation of the morphology of surface cracks.

2.3. Applied deformation experiments

Flat specimens were prepared by spark erosion with the loading axis aligned parallel to the rolling direction (RD). The specimens for monotonic deformation had a gauge length, width and thickness of 4 mm, 2 mm, and 1 mm, respectively. Corresponding dimensions for cyclic loading were 4 mm, 3 mm, and 2 mm, respectively. Monotonic tensile deformation was carried out at an instrument by Kammrath & Weiss GmbH, equipped with a DIC system (ARAMIS, GOM mbH). Deformation was conducted at room temperature at a constant cross-head speed of 5 μm s^{-1}. Fig. 2 shows the σ–ε curve of the utilized DP-steel, averaged over several measurements. One specimen was separately deformed up to the onset of necking, where four equal-sized areas were selected over the specimen surface for further damage investigation. The average strain level for these areas were determined by DIC as $\varepsilon_1 = 0.02$, $\varepsilon_2 = 0.07$, $\varepsilon_3 = 0.12$, and $\varepsilon_4 = 0.16$. The σ–ε curve for the applied – interrupted – deformation and the distribution of the analyzed areas are depicted in Fig. 2. Around 30 damage features were randomly selected within each area after mechanical silica OPS polishing. The features were captured by SE and ECCI, using a Zeiss Merlin SEM, and scanned by EBSD at a step size of 50 nm, using a JEOL JSM-6500F FE-SEM equipped with a TSL OIM system. The damage mechanisms were characterized, regarding their common microstructural configurations.

Regions of similar configurations were selected in an undeformed specimen, and tracked under interrupted uni-axial tensile deformation in order to verify the microstructural influence for each mechanism and to better resolve damage at the nucleation phase. The deformation proceeded in global elongation steps of 500 μm, corresponding to ε_{av} = 12.5% per step. Frames of FIB-milled marks were used for strain