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High resolution in situ mapping of microstrain and microstructure evolution reveals damage resistance criteria in dual phase steels

Dingshun Yan, Cemal Cem Tasan *, Dierk Raabe

Max-Planck-Institut für Eisenforschung GmbH, Max-Planck-Str. 1, 40237 Düsseldorf, Germany

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ABSTRACT

Microstructures of multi-phase alloys undergo morphological and crystallographic changes upon deformation, corresponding to the associated microstructural strain fields. The multiple length and time scales involved therein create immense complexity, especially when microstructural damage mechanisms are also activated. An understanding of the relationship between microstructure and damage initiation can often not be achieved by post-mortem microstructural characterization alone. Here, we present a novel multi-probe analysis approach. It couples various scanning electron microscopy methods to microscopic-digital image correlation (μ-DIC), to overcome various challenges associated with concurrent mapping of the deforming microstructure along with the associated microstrain fields. For this purpose a contrast- and resolution-optimized μ-DIC patterning method and a selective pattern/microstructure imaging strategy were developed. They jointly enable imaging of (i) microstructure-independent pattern maps and (ii) pattern-independent microstructure maps. We apply this approach here to the study of damage nucleation in ferrite/martensite dual-phase (DP) steel. The analyses provide four specific design guidelines for developing damage-resistant DP steels.

1. Introduction

The drive toward improved combinations of high strength and ductility motivates the design of novel alloys with complex, multi-phase micro-/nano-structures. Many of the recently introduced alloys demonstrate this microstructural complexity, containing multiple phases of different composition, crystallography, morphology, dispersion, stability and size. Examples are ultrafine-grained α/α' dual-phase (DP) steel [1], α'/γ transformation-induced plasticity (TRIP) steel [2], Triplex steel [3], TRIP-maraging steel [4], β/α titanium alloys [5] and α/X magnesium alloys [6]. Some of them are shown in Fig. 1. Such alloys present a composite-like micro-mechanical response, which in turn enables tuning optimal combinations of strength and ductility by adjusting the phase fractions as well their individual properties, interfaces and morphology.

However, incorporating phases of high mechanical contrast promotes the risk of micro-cracking at spots of high stress and/or strain mismatch. This in turn may cause early mechanical softening, or even catastrophic failure. Due to these reasons, damage evolution has been intensively studied in recent years especially in DP steels [7–13]. Most common damage sites in DP steels are the martensite/ferrite interfaces (**M**/**F**) or martensite island interiors (**M**) [9]. However, there are different views on exactly how these mechanisms nucleate and interact with each other. Kang et al. [10] and Avramovic et al. [7] both reported that the early-stage damage incidents are initiated inside **M** prior to percolative plastic instability, while other damage mechanisms are activated following such mesoscale localization phenomena. However, Avramovic et al. [7] also noted that **M**/**F** damage incidents play a more critical role for the overall properties. Maire et al. observed a more balanced damage activity of **M**/**F** and **M** [11]. Recently, Hoefnagels et al. have carried out an extensive analysis through quantitative characterization of the influence of the starting microstructure, strain path and strain level on the resulting damage mechanisms [9]. The obtained experimental and simulation results were explained in terms of a hypothesis that proposes that the two mechanisms are intrinsically coupled, i.e., the **M**/**F** damage incidents are typically initiated by **M** cracking.

These contradicting views arise from the insufficient resolution in the analysis of deformation and damage at the same position, with respect to their strong heterogeneity at microstructure-scale [8,12,13]. Thus, it is required to introduce novel analysis methods, which make use of advanced high resolution probing techniques of micro-mechanical processes during deformation. However, this is

* Corresponding author. Tel.: +49 211 6792 866; fax: +49 211 6792 333.
E-mail address: c.tasan@mpie.de (C.C. Tasan).

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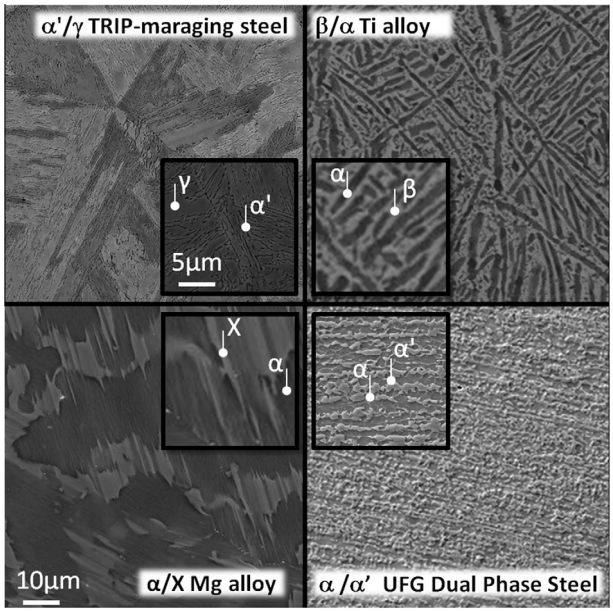

Fig. 1. Dual-phase microstructures of some recently introduced bulk nanostructured alloys: TRIP-maraging steel (courtesy of Meimei Wang), β/α titanium alloys (courtesy of Zahra Tarzimoghadam), α/X magnesium alloys (courtesy of Jinkyung Kim) and ultrafine-grained α/α′ dual-phase steel. Phases are marked in the zoomed-in insets.

an experimental task that imposes multiple challenges, as it requires the simultaneous mapping of the deformation-induced evolution of the (i) microstructure, (ii) microstrain and (iii) microstress fields at a representatively large field-of-view and yet, a sufficiently high spatial resolution.

Here, we present new insights into damage nucleation in DP steels by developing a novel methodology that overcomes challenges (i) and (ii).[1] This methodology combines in-situ scanning electron microscopy (SEM) testing with optimized microscopic-digital image correlation (μ-DIC) analysis. In this regard, the following report is divided into three parts. Part-A, "Methodology Development", presents a detailed overview of the currently existing approaches, to motivate the need for introducing an advanced methodology, and to point out the ingredients of the optimal strategy in that context. In Part-B "Proof-of-Principle", a detailed description and assessment of the introduced coupled microstrain and microstructure mapping (με & μS mapping) methodology is presented. Finally in Part-C, "Case Study", the developed method is applied to investigate damage evolution in DP steel with the final aim to identify guidelines for designing damage-resistant microstructures.

PART-A

2. Methodology development

2.1. Challenges in coupled με & μS mapping in SEM

For challenge (i), i.e. mapping deformation-induced microstructure evolution, SEM is the ideal observation tool due to two main reasons. First, it allows the operation of multiple imaging detectors

[1] The microstructure and microstrain fields obtained in the presented approach also enable a numerically-assisted indirect solution for challenge (iii), i.e. the associated stress field calculations. This aspect is discussed in detail elsewhere though [14].

Table 1
Imaging modes in SEM environment that allow investigation of different micro-mechanical phenomena.

Imaging Mode	Micro-mechanical phenomena
Secondary Electron	Damage mechanisms [10,19], slip trace analysis [20], surface roughening [21], shear banding [22], etc.
BackScattered Electron, Electron Channeling Contrast Imaging	Dislocation imaging [23,24], sub-grain formation [25,26], mechanical twinning [27,28], phase transformation [29], etc.
Electron BackScatter Diffraction	Micro-texture [30,31], phase transformation [32–34], defect density [35], sub-grain formation [36,37], slip trace analysis [38], etc.

that are capable of probing a set of relevant micro-mechanical phenomena (Table 1) at an optimal combination of spatial resolution and field-of-view. In this regard, recent developments in electron backscatter diffraction (EBSD) and electron channeling contrast imaging (ECCI) techniques are of specific significance, as they enable current SEM's to deliver quantitative, spatially-resolved mapping of crystallographic features and defects [15,16], even single dislocations [17]. Second, with respect to techniques that provide improvements in spatial resolution, e.g., transmission electron microscopy, or in 3D analysis capabilities, e.g., X-ray micro/nano-tomography [11,18], requirements on sample size, surface quality, and imaging are far less stringent. This flexibility strongly helps imposing well-defined deformation boundary conditions and implementation of a multi-probe imaging approach.

For tackling challenge (ii), i.e. mapping deformation-induced microstrain evolution, the recently introduced μ-DIC approach is the ideal route since it provides the most direct coupling to high resolution microstructure maps obtained during deformation [19,39]. DIC requires registering and correlation of a random pattern to calculate displacement fields and from these the corresponding strain maps [40,41]. While DIC is typically used with optical camera images [42–44], μ-DIC is based on images from a microscope. Principally image series from any microscope can be used for μ-DIC. However, considering the spatial resolution and field-of-view requirements, and the need for direct coupling to deformation-induced microstructure evolution, SEM based μ-DIC [19,45] excels as the ideal approach with respect to other alternatives, e.g., optical microscopy [46] and atomic force microscopy [47].

While SEM is identified as the ideal medium for microstructure or microstrain mapping, challenges arise when coupling them. These shortcomings are next summarized by categorizing the previous such efforts into two groups based on the nature of the DIC pattern on the sample, namely, using microstructure-based patterns on the one hand vs. artificial patterns on the other hand. For these groups, Fig. 2 schematically demonstrates the evolution of the pattern and the corresponding μ-DIC and EBSD maps at different deformation levels.

The first group relies on tracking specific microstructural features for correlation. Examples are slip traces in BSE [48] or SE [45] images, and especially boundaries in SE images of etched microstructures [10,19,49,50] or in EBSD image quality maps [51]. Owing to its practicality, this approach is the most popular microstrain mapping strategy, despite several, often overlooked, limitations: First, as pointed by arrow-1 in Fig. 2, etching causes considerable undesired microstructure manipulation, such as grain boundary grooving, that may alter the true strain field through local stress intensification. Second, as indicated by arrow-2 in Fig. 2, spatial strain resolution of these approaches is intrinsically coupled to the average grain size, and hence, is often insufficient to resolve in-grain strain heterogeneities. Moreover, as shown by