

Statistical analysis of damage in relation to strain-path,
microstructure and mechanical contrast

Von der Fakultät für Georessourcen und Materialtechnik der
Rheinisch-Westfälischen Technischen Hochschule Aachen

zur Erlangung des akademischen Grades eines

Doktors der Ingenieurwissenschaften

genehmigte Dissertation

vorgelegt von

Maximilian Aeneas Wollenweber, M.Sc.

Berichter: Univ.-Prof. Dr. Sandra Korte-Kerzel

Prof. Dr. C. Cem Tasan

Tag der mündlichen Prüfung: 19.02.2026

Diese Dissertation ist auf den Internetseiten der Universitätsbibliothek online verfügbar

Declaration on publications

This thesis includes the following research articles that have been published in international peer-reviewed journals:

Publication 1: *"On the automated characterisation of inclusion-induced damage in 16Mn-CrS5 case-hardening steel"* **Wollenweber, M.A.**, Kusche, C.F., Al-Samman, T. and Korte-Kerzel, S.

Advances in Industrial and Manufacturing Engineering, vol. 7, 2023
10.1016/j.aime.2023.100123 [1]

Publication 2: *"On the damage behaviour in dual-phase DP800 steel deformed in single and combined strain paths"* **Wollenweber, M.A.**, Medghalchi, S., Guimaraes, L., Lohrey N., Kusche, C.F., Kerzel U., Al-Samman, T. and Korte-Kerzel, S.

Materials & Design, vol. 231, 2023
10.1016/j.matdes.2023.112016 [2]

Publication 3: *"On the plasticity of MnS at elevated temperatures and its influence on damage prevalence"* **Wollenweber, M.A.**, Werner, J., Kusche, C.F., Tian, C., Sun, P-L., Gerlach, J., Al-Samman, T. and Korte-Kerzel, S.

Acta Materialia, vol. 291, 2025
10.1016/j.actamat.2025.120987 [3]

This thesis also includes the following pre-print of a research article that has been published online on RWTH Publications:

Publication 4: *"Ductile damage at MnS inclusions in 16MnCrS5 steel - influence of the phase environment"* **Wollenweber, M.A.**, Reclik, T., Reinhart, C., Maier-Kiener, V., Al-Samman, T., Kerzel U. and Korte-Kerzel, S.

RWTH Publications, 2025
10.18154/RWTH-2025-10405 [4]

Acknowledgments

Doing a PhD is tough. However, having such a supportive environment as I had during the last years makes it considerably easier. And in turn, this section fills up quickly, as I have so many people to thank for so many things.

First and foremost, I want to thank Sandra and Talal. It may sound cliché, but thank you for the opportunity. Thank you for the chance to conduct so many interesting experiments, for enabling me to learn new and highly specific things, for allowing me to interact with and teach material's science students (who were forced to listen to me talk about what I enjoy doing), all while being paid for it. I had a great time. But more importantly, thank you for your open doors, always being on my side, and our ever solution-oriented discussions.

At the same time, I also want to thank Cem for welcoming me into his lab for five months and taking the time to read this thesis and examine me in my defence. At the time of writing this, I am still very much looking forward to all the good times ahead in your lab. Much of the work that I present here would not have reached such quality or quantity without the help of my many student assistants and students who wrote their theses with me. Thank you Luiz, Nicole, Shiv, Elif, Dominik, Fadi, Malik, Clara and Sebastian. Out of these wonderful people I particularly want to thank Clara, you did amazing work over two theses! I also want to especially thank Dominik who supported me so much for the first years of my PhD. You never let me down and laid so much groundwork for everything that followed. Finally, the biggest thanks go to Fadi. I know you bent over backwards for our projects. I'm deeply grateful for all those long nights you spent working so this thesis could be completed. I also want to thank all my colleagues and former colleagues who were so patient with me while I was (and am) learning from them, and who remained supportive even in less scientific matters.

There is no particular order here but I'll start by thanking Carl: you were a huge mentor even before my PhD began! Thank you for finally accepting me as a Bachelor student and teaching me both how fun (and how serious) PhD life can be. I also want to thank Muhammad. You were my other major influence prior to my PhD. Not only did you jump-start my citation count by involving me in your excellent work, you also helped me appreciate things I'd taken for granted and gave me self-confidence through our scientific discussions. Thank you Tom. I'm so happy you came back for your PhD! Thanks for high-concentration sessions, way too in-depth side-topic discussions, and making me realise that yes, I can visualise an apple in my head and rotate it. Thank you Lukas. Thank you

for discussing basic materials physics concepts with me (and reminding there's always another level), plus all the jokes and word associations! Thank you Nina, for great E03 "Lehre Büro" times, gym sessions, mental support, reading this thesis and giving feedback, but most of all being a role model. Your hard work encouraged (and still encourages) me greatly. Thanks you Christina for all those nice PhD hats (with Nina) and especially dealing with "PhD defence 2.0" alongside me: it was far more fun than I'd thought possible! Thank you Malik, for bringing life back to E03, staying late in the office with me and taking over most non-scientific responsibilities.

Also thank you everyone else who shared lunch/laughter/frustration with me. In particular Setareh, Thomas, Lars, Mattis, Wassili and Nisa. Of course thanks too everyone who took administrative burdens off my shoulders, in particular Miriam, Gabi and Sergej, and everyone helping prepare samples or teaching machines: especially Thomas, David, Arndt, André and Detlef. I extend thanks too to all TRR colleagues with whom I've had great discussions that sometimes even led somewhere scientifically: Niklas, Jannik, Jan, Jonas, Doro, Martina, Konrad, Angelica, Robin, Philipp, Annemarie and Wan.

Outside of work: heartfelt thanks go out to my brother Alex and sister Lisa, you gave unwavering support through every stressful moment caused by or affecting academic life! The same holds true for my parents, who always believed in me and gave me so much freedom during this special time of life. Danke Mama und danke Papa! Finally, I want to especially thank Pia: always by my side at work and home alike. Your unlimited patience, ways of brightening any mood, unwavering support, all played a huge part in making this one of the greatest times of my life. Words cannot express how thankful I am.

Table of Contents

TABLE OF CONTENTS	I
LIST OF ABBREVIATIONS	III
ZUSAMMENFASSUNG	1
ABSTRACT	2
1 Motivation and Objectives	3
2 Materials & Methods	7
3 Fundamentals	9
3.1 Damage in metal forming	9
3.1.1 Definition of damage	9
3.1.2 Types of damage and damage initiation mechanisms	10
3.1.3 Interactions of damage with: strain state, microstructure, mechan- ical contrast	13
3.1.3.1 Stress state	13
3.1.3.2 Microstructure	16
3.1.3.3 Mechanical contrast	18
3.2 Key techniques for damage analysis	20
3.2.1 SEM based analysis techniques	20
3.2.1.1 Large scale, high-resolution SEM imaging	20
3.2.1.2 Electron backscattered diffraction	22
3.2.2 Measurement of constituents' mechanical properties	22
3.2.2.1 Nanoindentation	22
3.2.2.2 Micropillar compression	24
4 Key insights and outlook	27
4.1 Key insights	27
4.2 Outlook and future work	34
Bibliography	47
5 Publication 1	48

6	Publication 2	56
7	Publication 3	73
8	Pre-print of publication 4	88

List of Abbreviations

<i>SEM</i>	Scanning Electron Microscopy
<i>SE</i>	Secondary Electron
<i>EBSD</i>	Electron Backscatter Diffraction
<i>EDS</i>	Energy-Dispersive X-ray Spectroscopy
<i>DP</i>	Dual-Phase
<i>MnS</i>	Manganese Sulphide
<i>NoV</i>	Number of Voids per unit area
<i>VAF</i>	Void Area Fraction
<i>CSM</i>	Continuous Stiffness Measurement
<i>DAF</i>	Diamond Area Function
σ	normal stress components
τ	shear stress components
η	triaxiality
<i>L</i>	Lode parameter
<i>H</i>	hardness
<i>E</i>	elastic modulus

Zusammenfassung

Die Entwicklung fortschrittlicher Stähle und Umformprozesse mit reduzierter Umweltbelastung ist ein zentrales Ziel der modernen Materialwissenschaft. Diese Dissertation untersucht Strategien für das Schädigungskontrollierte Umformen, ein Ansatz, der darauf abzielt, die Materialeigenschaften durch minimale Änderungen an bestehenden Prozessen oder Werkstoffen zu verbessern. Im Fokus dieser Arbeit steht dabei die Analyse des Einflusses von Mikrostruktur, mechanischem Kontrast und Spannungszustand auf die Schädigungsnukleation und -entwicklung in Stählen, insbesondere in Dualphasen- (DP) und Mehrphasenstählen wie 16MnCrS5.

Es wurden zwei automatisierte Methoden zur Quantifizierung der Schädigungshäufigkeit mittels Rasterelektronenmikroskopie entwickelt: Eine nutzt die Lokalisierung von Einschlüssen, um eine effiziente Analyse in Stählen zu ermöglichen, bei denen Einschlüsse die Hauptursache für Schädigungen sind. Die andere Methode basiert auf der Segmentierung geätzter Mikrostrukturen, um detaillierte Korrelationen zwischen lokaler Mikrostruktur und Schädigungsentstehung zu erfassen. Diese Ansätze wurden am Stahl 16MnCrS5 validiert: Während die erste Methode durch größere Allgemeingültigkeit und Robustheit gegenüber Bildgebungsbedingungen besticht, liefert die zweite eine höhere Informationsdichte, allerdings nur für spezifische Mikrostrukturen.

Experimente zeigten, dass durch gezielte Variation von Lastpfaden und Belastungsrichtungen die Schädigungshäufigkeit signifikant reduziert werden kann. Beispielsweise führt eine Reihenfolge von Druck- vor Zugbelastung zur Verfestigung von Ferrit und verringert die Entstehung von Schädigung durch abnehmenden mechanischen Kontrast. Dessen Rolle im 16MnCrS5 wurde durch Versuche bei unterschiedlichen Temperaturen verdeutlicht: Unterschiedliche Verfestigungsraten der Phasen bei verschiedenen Temperaturen bestimmen die Inkompatibilität während der plastischen Verformung und beeinflussen so maßgeblich die Schädigungshäufigkeit innerhalb des Werkstoffs.

Untersuchungen der lokalen Phasenumgebungen in Mehrphasenmikrostrukturen zeigen, dass die Nukleation neuer Schädigungsstellen nicht signifikant von der unmittelbaren Umgebung abhängt, während das anschließende Wachstum dieser Stellen jedoch stark durch die umgebenden Phasen bestimmt wird. So wachsen Schädigungsstellen in MnS-Einschlüssen besonders stark, wenn sie vollständig von Ferrit umgeben sind, während eine vollständige Einbettung in Perlit das Wachstum deutlich hemmt. Diese Ergebnisse implizieren außerdem, dass Designprinzipien für schädigungstolerante Dualphasenstähle nicht direkt auf komplexere Mehrphasensysteme übertragbar sind.

Abstract

The development of advanced steels and forming processes with reduced environmental impact is a critical objective in modern materials science. This dissertation investigates strategies for damage-controlled forming, a strategy which aims to improve material properties with only minimal changes, by systematically analysing how microstructure, mechanical contrast, and stress state influence damage nucleation and growth in steels, particularly dual-phase (DP) and multi-phase alloys such as 16MnCrS5.

To this end, two complementary automated tools for quantifying damage prevalence using scanning electron microscopy were developed: one uses inclusion-based localization to enable high-throughput analysis in steels where inclusions drive damage formation. The other employs segmentation of etched microstructures to provide detailed correlations between local constituent arrangement and damage occurrence. These approaches were validated on 16MnCrS5 steel, revealing both their strengths and limitations: one offers greater generality and robustness to imaging conditions, while the other provides higher information density but is applicable only to more specific microstructures.

Experimental investigations demonstrated that strategic manipulation of strain paths and loading directions can significantly reduce damage prevalence. For example, sequencing compressive before tensile deformation was found to promote strain hardening in ferrite and minimise damage prevalence by reducing mechanical contrast. The role of mechanical contrast was also elucidated in 16MnCrS5 through temperature-dependent testing: non-linear relationships between constituent hardening rates at different temperatures, rather than yield strength alone, were shown to govern incompatibility during plastic deformation, thereby affecting overall material integrity.

A particular focus was placed on understanding how local phase environments affect damage behaviour in multi-phase microstructures. It was shown that while nucleation of new damage sites is not strongly dependent on local environment, subsequent growth is highly sensitive to the surrounding phases: MnS inclusions engulfed by ferrite experience greater void growth, whereas encapsulation within pearlite provides effective shielding. These findings challenge the direct transferability of dual-phase design principles to more complex alloys.

Chapter 1

Motivation and Objectives

Modern materials science research is strongly influenced by the need to design materials and processes that mitigate the negative impact of high energy consumption and greenhouse gases on the environment. In particular, the steel industry plays a critical role in anthropogenic climate change, as it accounts for a significant share of consumed energy and emitted greenhouse gases [5–9]. To reduce the environmental impact, numerous innovative and promising solutions have been proposed, such as the use of hydrogen-based direct reduction [10, 11], expansion of electric arc furnaces powered by renewable energy [12], and the implementation of carbon capture and storage systems [13]. While these approaches offer significant potential for reducing greenhouse gas emissions, they also require major shifts in industrial practice, including substantial financial investments and extended implementation timelines. In this regard, damage-controlled forming, which has gained more interest in recent years [14–17], offers a unique solution. This approach requires only minimal modifications to the existing forming process or material composition to enhance component performance and extend service life [18]. Additionally, more precise information about the time-point of failure can encourage re-evaluation of safety concepts with the goal of decreasing excess material in components. Consequently, modifications can be implemented quickly, with relatively low financial investments, while still leading to a decrease in material usage and emissions. The phenomenon of damage in metals is well established and has been the focus of extensive research in the past. Fundamental experimental work has elucidated the processes of damage nucleation, growth, and coalescence [19–21] while theoretical models have been formulated to capture their impact on a material’s mechanical behaviour [22]. Many investigations have employed Dual-Phase (*DP*) steels [23–25] and aluminium alloys [26–28] as representative model systems for studying damage mechanisms. However, linking these insights to forming processes and establishing design criteria that harness controlled damage to improve component performance remains a relatively young concept. Nonetheless, significant advances have been made in this direction from various perspectives. From a processing perspective, minor modifications in forming processes have been shown to reduce damage prevalence and enhance performance, for example by decreasing the triaxiality experienced by the workpiece during forming [18, 29]. From a simulation standpoint, emerging models now offer a more precise description of damage behaviour across different scales, thereby improving predictions of damage prevalence [30–32]. In general, these advances rely on

a comprehensive understanding of the fundamental damage mechanisms, which in turn necessitates investigation of damage in the material on a microscopic scale. In particular, such investigations lay the ground work for the physical description of a material's damage response under various conditions, as well as serving as a link between forming processes and their simulation. This connection is crucial, because while final component performance is quantified by mechanical properties (cf. [18]), simulated damage is generally represented by a scalar value [33, 34], making localised comparison of damage difficult. However, the result of both damage quantifications is rooted in physical processes which at their core involve the nucleation and growth of microscopic damage sites as well as their interaction with each other and external influences. Consequently, linking performance measurements to simulated damage and confirming damage predictions based on a physical understanding of damage, requires microscopic observations of damage sites as well as their quantification with respect to variation of external influences. Such damage sites are typically found in the form of voids in the microstructure and can be made visible by Scanning Electron Microscopy (*SEM*) for example [35]. Given the multitude of external factors, however, initial studies focused on developing a method for time-efficient damage quantification in *DP* steels, while simultaneously generating scale-bridging insights from individual damage site behaviour to component level distributions [36]. Despite marking significant advances toward damage-controlled forming, these studies primarily concentrated on basic strain path parameters such as applied strain and triaxiality [37]. However, key factors that could significantly advance damage-controlled forming in industrial practice have not yet been studied. In this study, we investigate more complex strain path parameters, diverse microstructures, and the mechanical contrast among constituent phases, which are key aspects that promise to bridge the gap between model-based and industrially viable damage-controlled forming [38–41]. In this context, more complex strain path parameters capture for example drastic changes in loading mode and direction, as observed for the combination of two simple strain paths conducted consecutively. Furthermore, differences in the mechanical properties of individual constituents and their change across temperature and strain-rate variations are incorporated through the quantification of mechanical contrast. Conversely, the study of different microstructures centres on the diverse geometric distributions of microstructural constituents and associated damage sites. Moreover, this study goes beyond the extensively characterised dual-phase microstructure by incorporating a second steel that exhibits a distinctly different microstructure. To this end, this work focuses on a fundamental understanding of the origins and evolution of microscopic damage sites while strategically varying these parameters. The key insights derived from this work address the following research questions:

1. *How can automated quantification of damage be achieved in steels exhibiting inclusion-induced damage?* ([Publication 1](#) & [Publication 4](#))
2. *How can strain path combinations and loading direction be used to strategically control damage prevalence through manipulation of stress state?* ([Publication 2](#))
3. *How does mechanical contrast affect damage prevalence, and how can processing parameters be adjusted to exploit this effect?* ([Publication 3](#))
4. *How do local phase environments affect damage prevalence in multi-phase microstructures?* ([Publication 4](#))

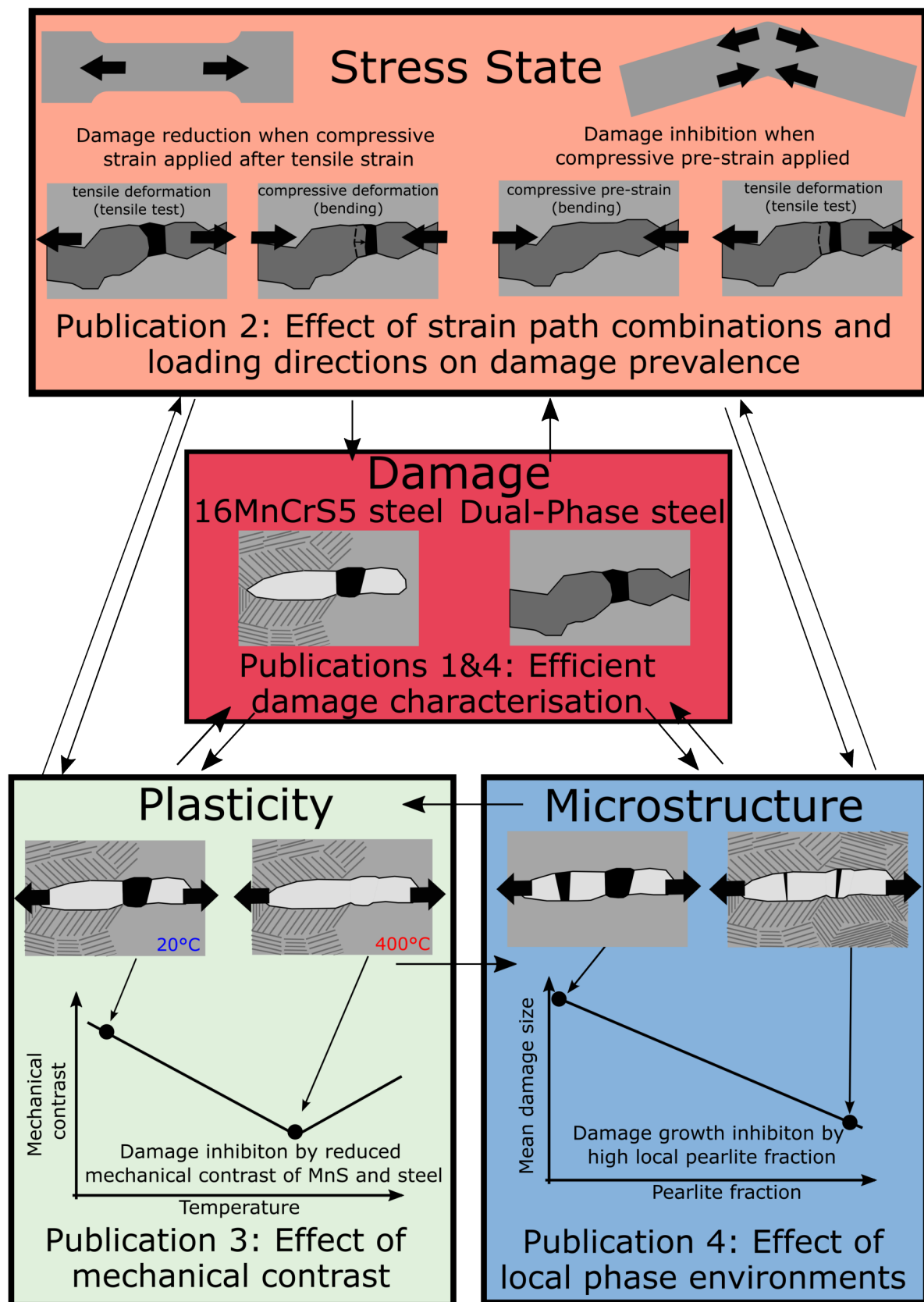


Figure 1.1: Schematic workflow of the interconnectivity between research questions and insights for the goal of damage controlled forming gathered in this dissertation.

Chapter 2

Materials & Methods

In this work, steel grades with different chemical compositions were analysed. The first material is a *DP* steel of DP800 grade, and the second is a case-hardening 16MnCrS5 steel. The chemical compositions of both steels are provided in Table 2.1 and Table 2.2:

Table 2.1: Chemical composition of 16MnCrS5 case-hardening steel in wt. % reprinted from [42].

Element	C	Si	Mn	P	S	Cr	Mo
Wt. %	0.20	0.17	1.24	0.03	0.03	1.05	0.04
Element	Ni	Al	Co	Cu	Nb	V	Fe
Wt. %	0.14	0.03	0.01	0.16	0.01	0.01	Bal.

Table 2.2: Chemical composition of *DP* steel in wt. % adopted from [40].

Element	C	Si	Mn	P	S	Al
Wt. %	0.131	0.194	1.69	0.0088	0.0016	0.038
Element	Cr	Mo	Nb	Ti	V	B
Wt. %	0.718	0.0035	<0.001	0.0303	0.0027	<0.001

The investigation of the microstructure, mechanical properties and damage of these materials was conducted using various analytical techniques. To induce deformation in samples and measure macroscopic mechanical properties, bending tests and tensile tests were employed. For analyses of micromechanical properties, microstructure, and damage sites, all samples were first metallographically prepared to ensure a plane and scratch-free surface. In cases where it was necessary, the microstructural constituents of the steels were revealed using nital. Afterwards, *SEM* micrographs, acquired using a Secondary Electron (*SE*) detector, were used to gather information about the microstructure and the damage distribution. For statistical evaluation regarding number and size of damage sites, *SE* micrographs typically covered large areas (approx. 1 mm²) and were analysed using machine-learning-assisted tools. *SEM*-based methods also included Energy-Dispersive X-ray Spectroscopy (*EDS*) scans to determine local chemical composition as well as Electron Backscatter Diffraction (*EBSD*) analyses to assess local crystallographic orientation. Micromechanical testing of individual constituent phases was conducted using nanoindentation, spherical nanoindentation, rapid nanoindentation mappings, as well as

micropillar compression experiments. The gained insights into microstructural characteristics, micromechanical properties, and strain paths were combined with measured damage distributions to infer their effects on damage prevalence.

Chapter 3

Fundamentals

3.1 Damage in metal forming

3.1.1 Definition of damage

Damage in the context of metal forming is not clearly defined, and its description is known to vary across different perspectives and research focuses [43–46]. Even when focusing on experimental studies, the definition of damage changes based on the type of experiment used for quantification [47], as shown in Figure 3.1.

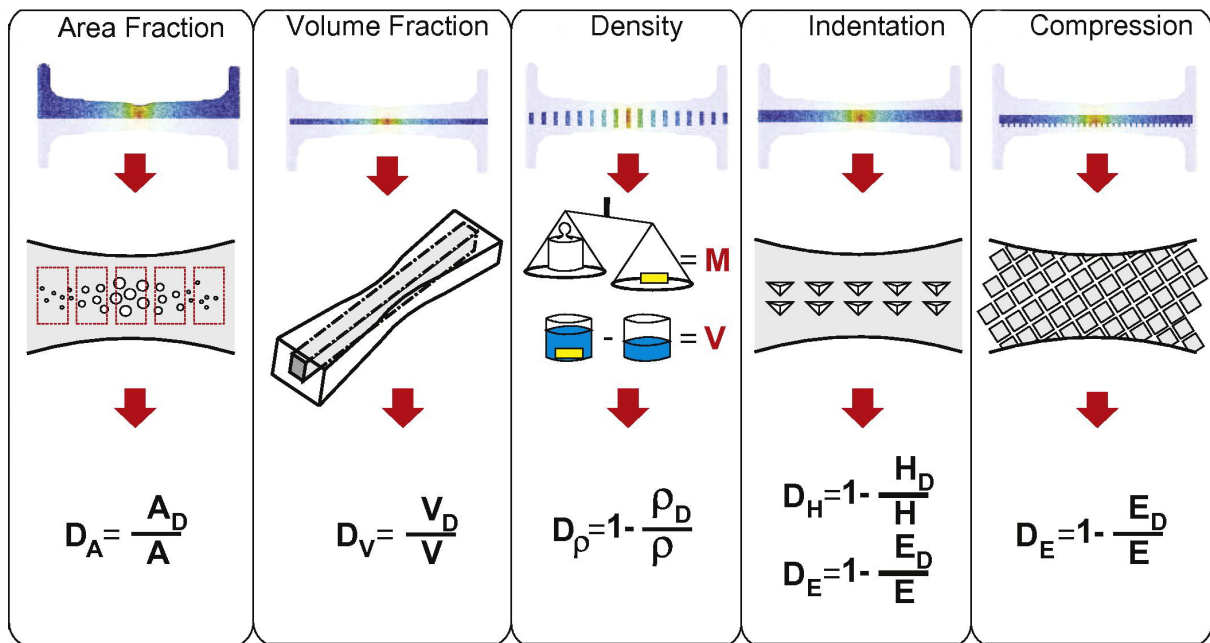


Figure 3.1: Varying definitions of the damage parameter in dependence on the experiment conducted for quantification. Adapted from [47] with permission from Elsevier.

From the perspective of this thesis, the relevant aspects of damage encompass its negative impact on mechanical properties, as well as its appearance, mainly characterised by microstructural voids. In accordance with these principles we adhere to a definition of damage given by Hirt et al. [48].

"Ductile damage causes the degradation of physical properties, in particular of mechanical properties, due to microstructural defects. These defects are characterised by the formation of new surfaces. In structural materials voids and micro cracks initiate

mostly by decohesion at interfaces such as grain boundaries and inclusions or due to brittle fracture of hard phases. The evolution of voids and micro cracks then causes damage under thermo-mechanical loading." [48]

Based on this definition, in this work, "damage prevalence" refers specifically to the quantification of both the number and size of microstructural damage sites. However, both the absolute number and total area covered by damage directly depend on the observed area. This dependence makes comparisons with other measurements obtained from areas of different sizes difficult. To counteract this effect, both parameters are typically normalised by the observed area. This leads to two key metrics used in this work: Number of Voids per unit area (NoV) and Void Area Fraction (VAF), which is the ratio between total void area and observed sample area. In some cases different normalization approaches are chosen where appropriate. For example inclusions-induced damage can be normalised to the total inclusion area. However, in all cases presented here both NoV and VAF are provided.

3.1.2 Types of damage and damage initiation mechanisms

As stated in the definition of damage given in subsection 3.1.1, damage sites are typically caused by microstructural heterogeneities. Since numerous types of defects can cause such heterogeneities in metallic microstructures, there exist various damage initiation mechanisms encompassing specific causes for the nucleation of a damage site. As an initial classification, we propose dividing damage mechanisms by the number of phases involved in the microstructure as illustrated in Figure 3.2.

Firstly, in single phase materials, the possible origins of damage sites are mainly limited to nucleation at grain boundaries as observed for example in polycrystalline copper [49] (cf. red box Figure 3.2). In dual-phase materials, however, additional mechanisms exist by which damage sites can form. In particular, interfaces between the two phases present a weak-point susceptible to the formation of voids [50] visualised in the green box of Figure 3.2. Furthermore, phases with different mechanical properties are often combined at the microstructural level to achieve favourable macroscopic behaviour. This frequently implies that one phase is more brittle than another and thus enables void formation following its fracture [51, 52]. Naturally, damage initiation at grain boundaries remains an active damage mechanism for dual-phase microstructures and may even be enhanced under certain conditions, as seen in some aluminium alloys [53]. Lastly, multiphase materials containing more than two constituent phases exhibit several types of interfaces. Depending on each phase's brittleness, fracture may not be restricted solely to the most brittle phase, as shown by the blue box in Figure 3.2. This results in an increased variety of possible initiation mechanisms [54].

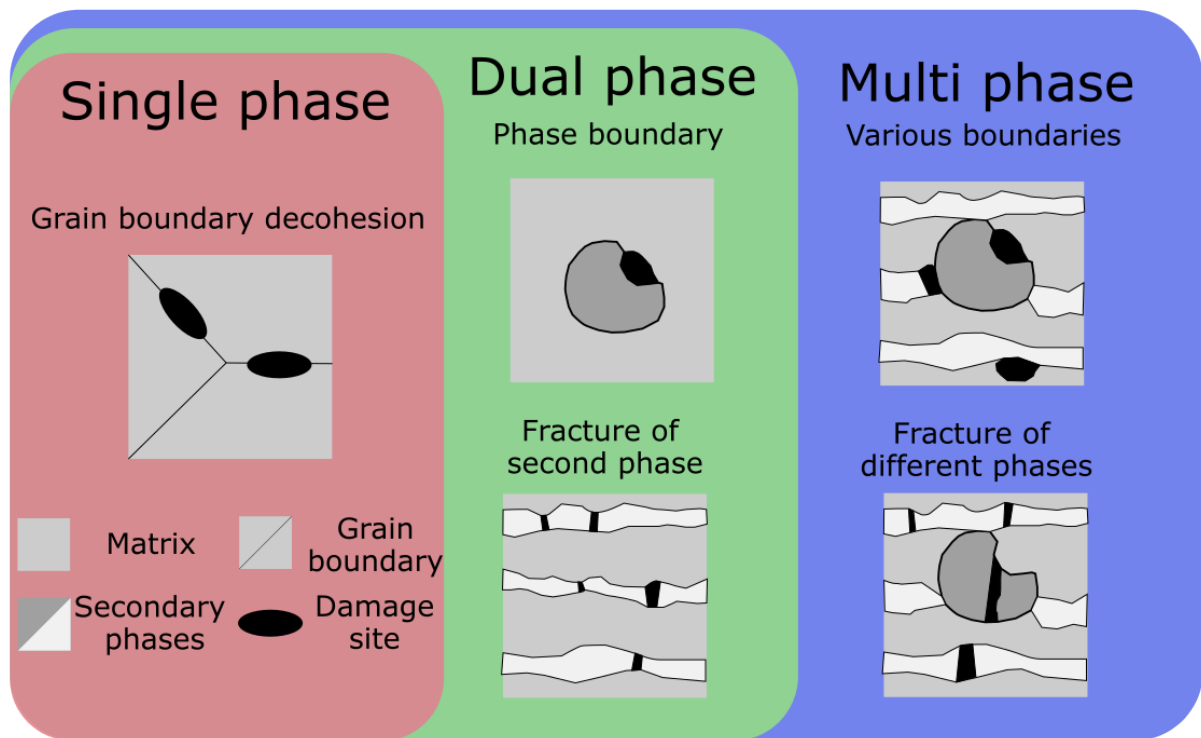


Figure 3.2: Classification of damage initiation mechanisms based on the number of microstructural constituents.

DP steels, as the name suggests, are a prominent example of the dual-phase material category and harbour a microstructure composed of a ferritic matrix and martensite, which is comparatively stronger but more brittle [40, 55]. Depending on processing, the martensitic phase can assume various morphologies affecting mechanical properties and damage prevalence [56–58]. The damage mechanisms in *DP* steel are well explored and encompass the nucleation of voids at ferrite–ferrite grain boundaries, ferrite–martensite interfaces, and due to fracture of the martensitic phase [23, 41]. *SEM* micrographs illustrating these mechanisms are shown in Figure 3.3.

Although these mechanisms are seemingly unambiguously defined in literature, the typical morphology of damage sites observed in *DP* steel using *SEM* can be less clear, owing to their three-dimensional nature. Serial sectioning experiments have shown that the underlying cause of void nucleation may be obscured in *SEM* images due to their two-dimensional nature. Consequently, a single damage site can appear to result from different initiation mechanisms when observed on different two-dimensional sections of the microstructure [59].

16MnCrS5 steels do not belong to the class of dual-phase microstructures but rather fall under multiphase materials. In this case, a dual-phase microstructure composed of

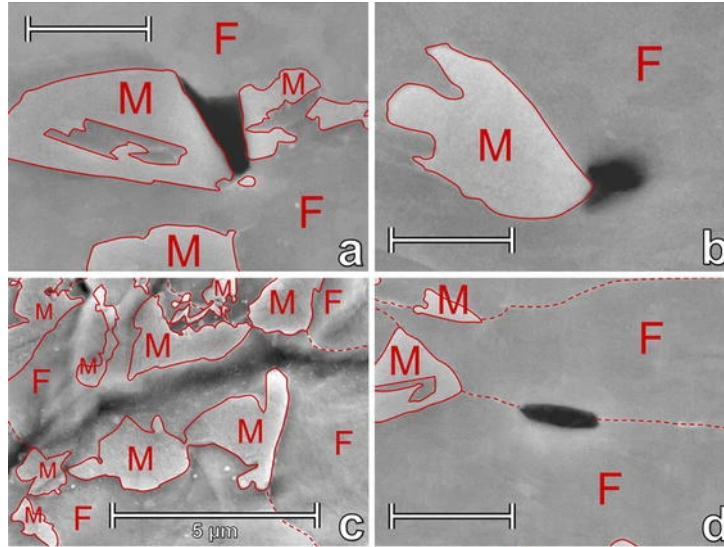


Figure 3.3: SEM micrographs of damage mechanisms in *DP* steels. M and F correspond to martensite and ferrite accordingly, while dotted lines indicate grain boundaries. a) Fracture of martensite b) and c) decohesion at the M-F interface d) decohesion at ferrite grain boundary. Adapted from [41]. The original article was distributed under the terms of the Creative Commons Attribution 4.0 International License (<http://creativecommons.org/licenses/by/4.0/>).

ferrite and pearlite is complemented by Manganese Sulphide (MnS) inclusions. These inclusions are primarily responsible for damage nucleation [35, 60], similar to martensite in *DP* steels. MnS inclusions contribute to the damage prevalence either through fracture or by promoting void formation at their interfaces. In contrast to *DP* steel, however, assigning specific damage mechanisms is less ambiguous due to distinct morphological differences between damage sites occurring within or near MnS inclusions. In 16MnCrS5, however, damage is not limited solely to MnS , but damage sites within the steel matrix itself have also been observed [54], although with significantly lower prevalence compared to MnS -induced damages. The three-dimensional analysis conducted in [54] highlights the importance of inclusion-induced damage within 16MnCrS5 steels. However, so far it remained unclear how local environmental factors or surrounding phase distributions influence stress states within MnS inclusions and thus affect damage nucleation and evolution. In addition to mechanisms of initiation, the evolution of individual damage sites as a function of strain also typically serves as a criterion for classification. In general, a void progresses through three stages during deformation leading up to material failure as visualised in Figure 3.4 [61, 62].

Initially, in an ideal pristine material, there are no damage sites present. However, plastic deformation combined with mechanical heterogeneities leads to the formation of damage sites as the initial stage of their development. This process is often referred to as nucleation. At this point, the previously discussed initiation mechanisms can be most clearly identified. With increasing strain, damage sites can grow until their growth becomes

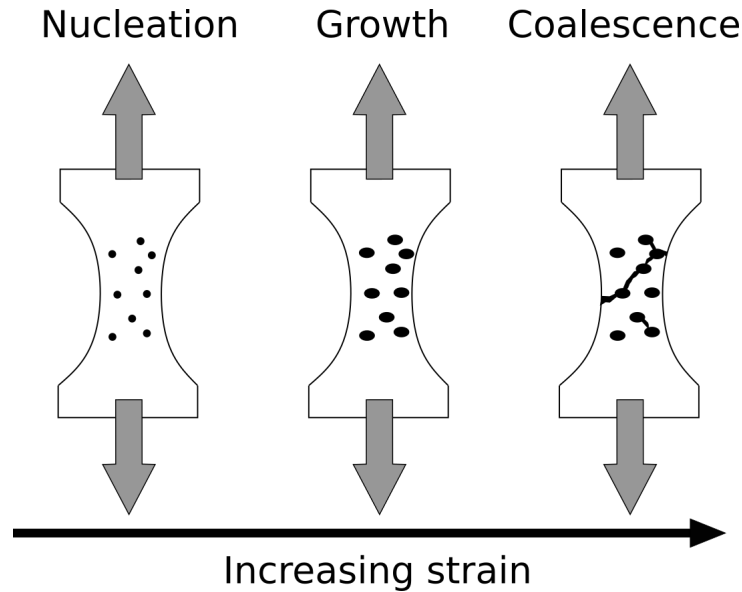


Figure 3.4: Stages of life of damage sites nucleated in metals deformed at positive triaxiality. Redrawn based on [61] with permission from Elsevier.

unstable due to coalescence with neighbouring damage sites. This ultimately leads to macroscopic crack formation and failure of the material [63–65].

3.1.3 Interactions of damage with: strain state, microstructure, mechanical contrast

Previous research on damage-controlled forming has identified key factors that influence damage prevalence during plastic deformation. Although these findings can be grouped from various perspectives, in this work they are organised according to the primary insights derived in this thesis: strain state, microstructure characteristics, and mechanical contrast.

3.1.3.1 Stress state

The stress state that a material experiences during forming is widely recognised in the literature as the primary influence on damage prevalence [66–68]. It forms the basis for many numerical models describing damage evolution and its effect has also been experimentally confirmed, including in studies of 16MnCrS5 [35] and *DP* steels [37]. The stress state itself can be described by a stress tensor, which consists of all stresses acting on a material during forming. In general, a distinction is made between normal stress components (σ) and shear stress components (τ), which together constitute the tensor shown below [69]:

$$\boldsymbol{\sigma} = \begin{bmatrix} \sigma_{xx} & \tau_{xy} & \tau_{xz} \\ \tau_{xy} & \sigma_{yy} & \tau_{yz} \\ \tau_{xz} & \tau_{yz} & \sigma_{zz} \end{bmatrix}$$

Here, the components satisfy the symmetry conditions:

$$\tau_{xy} = \tau_{yx}, \quad \tau_{xz} = \tau_{zx}, \quad \tau_{yz} = \tau_{zy}$$

These components depend on how the coordinate system is defined. The stress tensor can be transformed into its principal axis form so that it contains only normal stresses [69], as shown below:

$$\boldsymbol{\sigma} = \begin{bmatrix} \sigma_1 & 0 & 0 \\ 0 & \sigma_2 & 0 \\ 0 & 0 & \sigma_3 \end{bmatrix}$$

In this form, these normal stresses are referred to as principal stresses. The strains and shears induced as a result by these applied stresses can similarly be represented using a strain tensor [69]:

$$\boldsymbol{\varepsilon} = \begin{bmatrix} \varepsilon_{xx} & \varepsilon_{xy} & \varepsilon_{xz} \\ \varepsilon_{xy} & \varepsilon_{yy} & \varepsilon_{yz} \\ \varepsilon_{xz} & \varepsilon_{yz} & \varepsilon_{zz} \end{bmatrix} = \begin{bmatrix} \varepsilon_1 & 0 & 0 \\ 0 & \varepsilon_2 & 0 \\ 0 & 0 & \varepsilon_3 \end{bmatrix} \quad (\text{for principal stresses})$$

Here ε_{xx} , ε_{yy} and ε_{zz} represent normal strain components and ε_{xy} , ε_{xz} and ε_{yz} correspond to shear components. The applied strain acts as a direct driving force for damage nucleation, as has been observed experimentally in *DP* steel [37]. Typically, the number of damage sites increases exponentially with increasing strain, as visualised in Figure 3.5 a) for *DP* steel.

However, forming operations typically aim to achieve a specified geometry, which necessitates reaching a certain final strain. For this reason, adjusting the final strain is not considered an adjustable parameter for damage-controlled forming in this work. The manner in which that strain is achieved, on the other hand, depends on the configuration of the stress tensor during forming and can serve as a tool to control damage prevalence. Since the relationship between individual components of the stress tensor and damage prevalence is not always intuitive, it is common in continuum mechanics to use additional parameters to characterise complex stress states. These parameters can be especially valuable when analysing or predicting damage behaviour. The parameters most commonly encountered are the triaxiality (η) and the Lode parameter (L) [65] which are defined as follows [70]:

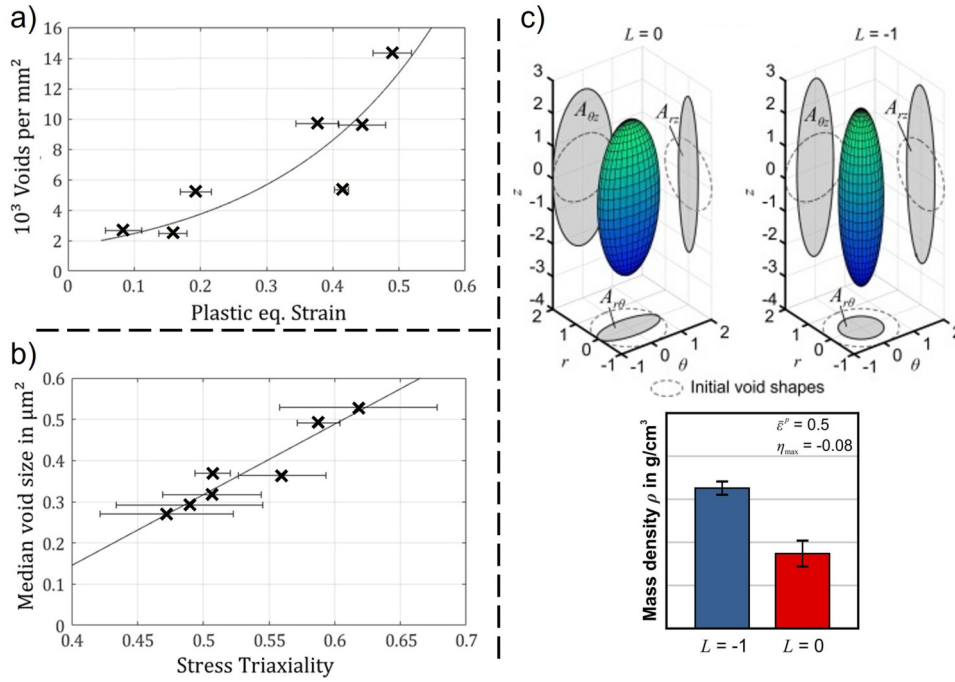


Figure 3.5: Influences of Strain, triaxiality and Lode parameter on damage prevalence. a) shows the exponential relationship between plastic equivalent strain and the number of damage sites in *DP* steel adapted from [37]. b) Shows the increase of median void area with increasing triaxiality. a) and b) adapted from [37] with permission from Elsevier. c) Shows the change of morphology of a damage site in 16MnCrS5 after plastic deformation with a Lode parameter of $L = 0$ and $L = -1$, as well as component's damage prevalence formed under corresponding stress states given by mass density measurements. c) Adapted from [70] with permission from Elsevier.

$$\eta = \frac{\sigma_1 + \sigma_2 + \sigma_3}{3\sigma_{eq}} = \frac{\sigma_h}{\sigma_{eq}}$$

$$L = \frac{2\sigma_2 - \sigma_1 - \sigma_3}{\sigma_1 - \sigma_3}, \text{ with } \sigma_1 \geq \sigma_2 \geq \sigma_3$$

Here, σ_h is the hydrostatic stress, σ_{eq} is the von Mises equivalent stress and σ_1 , σ_2 and σ_3 correspond to σ_{xx} , σ_{yy} and σ_{zz} from the above stress tensor, respectively. While their mathematical derivation may initially seem non-intuitive, their relationships with damage size and shape can be understood more readily. Triaxiality measures the ratio of volume-changing stresses to shape-changing stresses. A positive triaxiality indicates that stresses within a material, specifically at damage sites, act in an expansive manner, leading to increased void volume during deformation. This is visualised for *DP* steels in Figure 3.5 b). For negative triaxialities, a compressive stress state prevails, and the volume of damage sites is expected to decrease. At triaxialities below a certain cut-off value, void-driven ductile fracture is not expected at all [71]. Therefore, strategies aimed at decreasing overall triaxiality have proven as effective in reducing damage prevalence as they are intuitive. For both *DP* steel and 16MnCrS5 steel, specific modifications in forming processes have been demonstrated that reduce triaxiality at critical locations

during typical operations. For 16MnCrS5 steel, decreasing the shoulder opening angle during extrusion led to lower triaxialities, which was accompanied by decreased damage prevalence and improved performance [18, 35]. In *DP* steel, applying counter-pressure during bending achieved similar results and significantly reduced damage prevalence [29]. The second key parameter, the Lode parameter, describes variations in deviatoric stress states ranging from uniaxial tension through simple shear to uniaxial compression. It primarily influences void morphology rather than size [72]. However, even under constant positive triaxiality, variations in this parameter can still affect both damage prevalence and mechanical properties such as fracture strain [70]. In Figure 3.5 c), simulated changes in void morphology are shown for two extrusion processes differing only by their Lode parameters. The figure also demonstrates how these morphological changes influence overall damage prevalence, as determined via mass density measurements: a Lode parameter of $L = -1$ results in decreased observed damage prevalence. Note that here, a reduction in mass density signals an increase in total damage site volume.

3.1.3.2 Microstructure

The microstructure of a material refers to the arrangement of its constituents on a scale below the resolution of the human eye. Gottstein [69] defined the microstructure as:

"The arrangement of the constituents of a material, i.e. the spatial distribution of elements, phases, orientations, and defects are subsumed under the term microstructure." [69]

This is a broad definition, encompassing geometric distributions across different length scales, from larger phases on the order of millimeters down to atomic-scale defects such as vacancies. In this work, however, a more focused definition is adopted in order to better highlight which microstructural parameters can be adjusted to control damage prevalence. To this end, in this work microstructure is defined as the spatial distribution of constituents (including both individual phases and composite structures such as pearlite) and their grains within the material. A clear distinction is made between the geometric aspects of constituents, which are contained in this definition, and the mechanical properties of these constituents, which are not accounted for in this definition. Regardless of its precise definition, controlling microstructure to tailor mechanical properties remains fundamental in materials design, irrespective of underlying material system or processing route [73–75]. Even though, the influence of microstructure on damage evolution has received comparatively less attention in research, several key concepts to reduce damage prevalence have emerged. These concepts typically focus on dual-phase systems to identify optimal grain/phase size, morphology, and overall phase fraction. It has been shown,

for example, that in *DP* steel, smaller phases and grains, evenly distributed throughout an otherwise unconnected matrix, lead to reduced martensite fracture and less decohesion at interfaces [25, 56, 76, 77]. It was also shown that when combining two different phases of steels in a more ordered way than it is possible in monolithic *DP* steel, for example by roll bonding, a periodic arrangement of bands can even be beneficial for mechanical properties, when loading parallel to these bands [78]. The key insights gained from these studies demonstrated that damage and, by extension, mechanical properties affected by damage, are strongly influenced by how stress and strain distribute within the microstructure, which in turn is determined by the geometric arrangement of constituents. Designing a microstructure in which the comparatively more brittle constituents are finely and uniformly dispersed, and preferably have a spherical shape, tends to reduce overall damage. This is because such arrangements promote a more homogeneous distribution of stress and strain.

The effects of varying microstructural configuration on damage prevalence are illustrated in Figure 3.6. Here, *DP* steel with smaller microstructural constituents (Figure 3.6 a)) exhibited a slower evolution of damage as a function of strain compared to *DP* steel with larger constituents (Figure 3.6 b)).

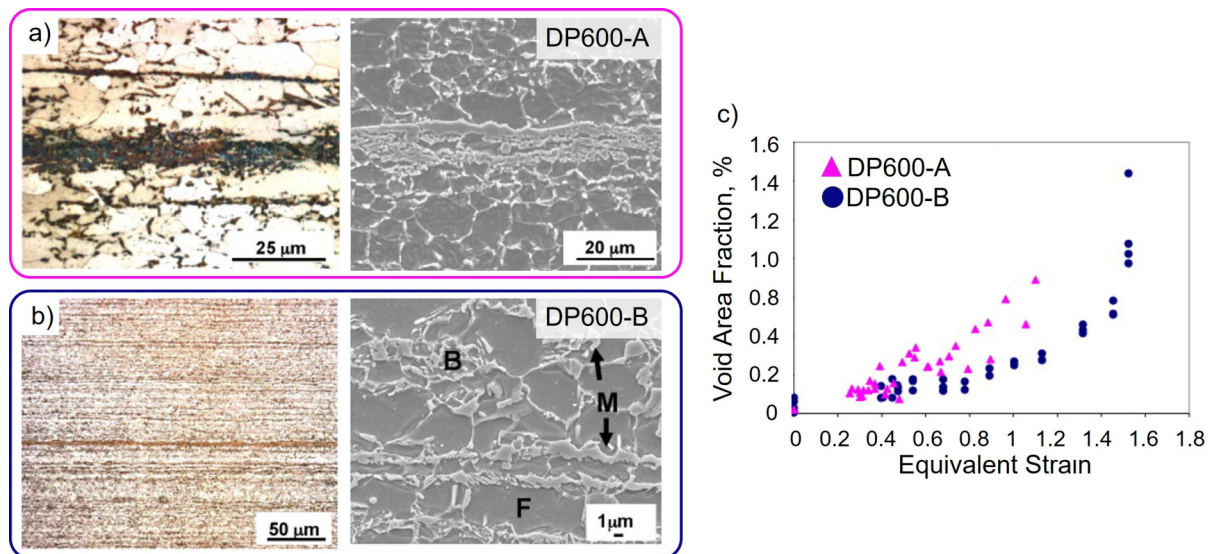


Figure 3.6: *DP* steel microstructures with microstructural constituents of different sizes. In a) martensite and ferrite phases are comparatively larger while in b) phases are comparatively smaller. c) shows the evolution of void area fraction as a function of the equivalent strain for both microstructures. Here DP600-A corresponds to a) and DP600-B corresponds to b). Adapted from [25] with permission from Elsevier.

These findings provide clear guidance for identifying and designing promising microstructures aimed at enabling effective damage-controlled forming in *DP* steels. However, whether this strategy will yield similar benefits when applied to multiphase materials remains questionable. For example, if constituents responsible for initiating damage have

higher melting points or other processing constraints, achieving an even distribution may be difficult, or even undesirable. In such cases, finely distributed particles could actually undermine efforts toward achieving damage-tolerant forming behaviour.

3.1.3.3 Mechanical contrast

Even though constituent properties were excluded from the definition of microstructure in the previous section, it is clear that the physical processes leading to damage nucleation and growth depend on the mechanical properties of all participating phases. As such, damage prevalence and its evolution with increasing strain are also influenced by these properties [79].

While absolute constituent properties are crucial for determining global mechanical performance and have been thoroughly studied in various *DP* steels [80, 81] in the context of failure and damage it is often their disparity that becomes most important. The difference in how each constituent participates in plastic deformation, along with resulting stress and strain distributions, has been widely discussed in literature as a key factor influencing damage initiation [82–84]. To describe this disparity between constituent mechanical properties, we use the term mechanical contrast in this work. The prevailing consensus is that reduced mechanical contrast generally benefits formability, a trend demonstrated for *DP* steels (see Figure 3.7) [79]. Understanding and optimising mechanical contrast can thus inform heat treatment protocols or compositional adjustments aimed at improving formability.

One simple way to quantify mechanical contrast among microstructural constituents is to calculate either their hardness difference (cf. Figure 3.7) or hardness ratio [85]. However, this simple approach does not always accurately predict damage prevalence [86]. For instance, increasing ferrite hardness versus decreasing martensite hardness can involve fundamentally different mechanisms potentially leaving critical configurations for damage initiation unchanged when only ferrite hardening occurs, whereas tempering martensite can more effectively suppress such sites. Furthermore, this ratio does not account for changes in phase hardness as a function of strain, a factor which has also been shown to significantly influence damage evolution. In this context, previous research indicates that *DP* steels containing ferrite with higher strain hardening capacity, which brings its behaviour closer to that of martensite, exhibit reduced damage prevalence compared to other *DP* steel variants [87]. Extending these concepts to multiphase materials remains relatively unexplored, likely due to increased complexity arising from additional phases (which complicates stress/strain distribution analysis) and because damaging phases like those found in *DP* steels may be less prevalent. Although some studies have characterised individual constituent phases such as *MnS* [88–90], data is less consistent. This is espe-

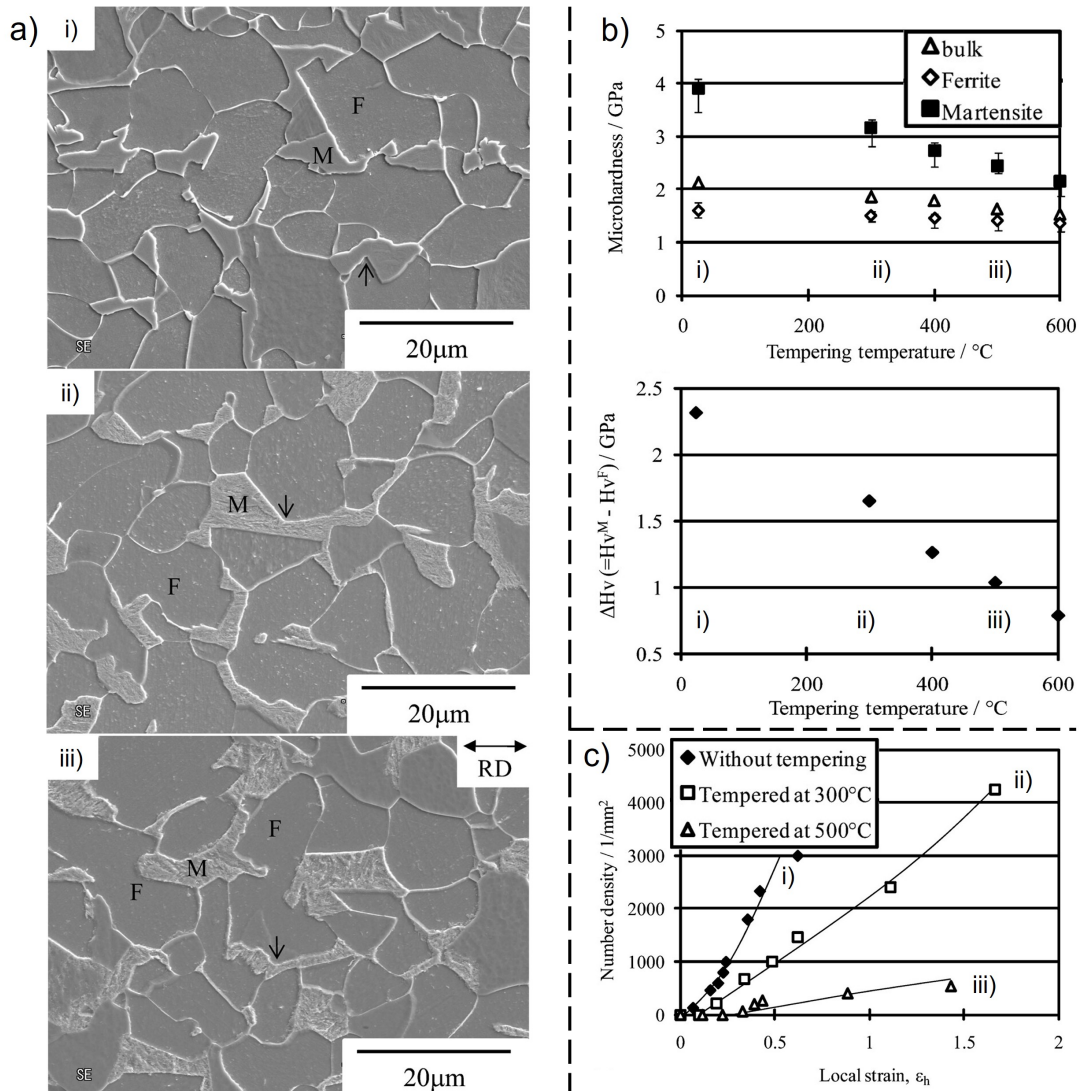


Figure 3.7: a) Dual-Phase steel microstructures with microstructural constituents of similar sizes without tempering (i), tempered at 300 $^{\circ}\text{C}$ (ii) and 500 $^{\circ}\text{C}$. b) microhardness and mechanical contrast between ferrite and martensite as a function of tempering temperature. The resulting microstructures from a) are highlighted. c) Damage prevalence given by number density in martensite phase as a function of local strain for the microstructures shown in a). Adapted from [79] with permission from Sage Journals.

cially relevant under more complex conditions such as for example increased temperature or strain-rates. Nevertheless, the principal strategy, to design microstructures promoting homogeneous stress/strain fields, is still expected to benefit multiphase materials.

3.2 Key techniques for damage analysis

3.2.1 SEM based analysis techniques

3.2.1.1 Large scale, high-resolution SEM imaging

It is clear that damage nucleation is strongly dependent on the local microstructure, which can vary significantly across samples, making nucleation inherently a statistical process. To ensure results are statistically meaningful and comparable, it is necessary to analyse sufficiently large areas. Defining what constitutes a sufficiently large area can be challenging and depends on the material. However, for microstructural segmentation of *DP* steel, it has been shown that even square images with side lengths exceeding 300 μm exhibit significant scatter when determining phase fractions [91]. This suggests that areas on the order of square millimetres are required for robust statistical damage analysis. In contrast, individual damage sites occupy much smaller regions, typically only a few hundred square nanometres, necessitating high-resolution imaging for accurate detection and quantification. Generally, capturing large areas at high resolution is time-consuming, especially given the vast number of damage sites present within each image. While automated acquisition simplifies image collection, the sheer volume makes manual identification and evaluation impractical within reasonable time frames. To counteract this, previous work [36, 92] has developed a method for automated detection and evaluation of damage sites. The developed method enables the workflow illustrated in Figure 3.8:

In this method thresholding and clustering techniques are employed to identify potential damage sites within the microstructure before classifying their initiation mechanisms using artificial intelligence. Because the networks used for classification of damage initiation mechanisms require training to be accurate, the application of this algorithm is limited to the specific *DP* steel originally studied. However, as the same *DP* steel was investigated in this work, the workflow could be directly applied throughout relevant portions of this study. Despite accelerating overall analysis [36], image acquisition itself, even when automated, remains time-consuming in particular for large areas due to high required resolution. Current research aims to further decrease imaging time, for example by employing additional machine learning networks to enhance image resolution. After acquiring images at lower resolution and performing resolution enhancement, features are detected and only the areas of interest are rescanned at high resolution [93].

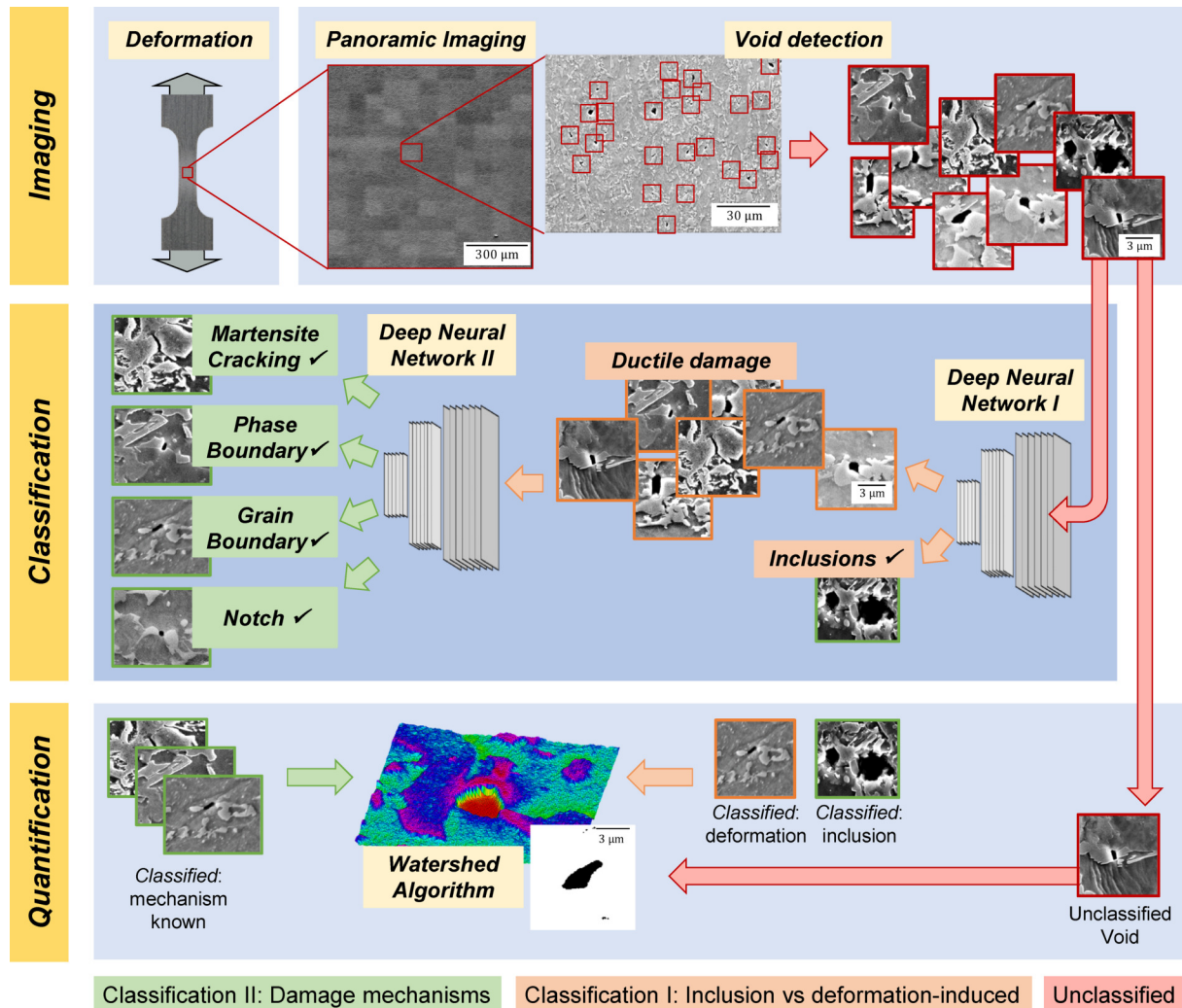


Figure 3.8: Workflow of damage analysis in *DP* steel featuring automated damage analysis. Sample deformation is followed by panoramic imaging and subsequent void detection by thresholding. Detected voids are then classified by two networks into inclusion-induced, or into one of several ductile damage initiation mechanisms. All detected voids independent from their classification result are also quantified by measuring their area. Reprinted from [36]. The original article was distributed under the terms of the Creative Commons Attribution License.

3.2.1.2 Electron backscattered diffraction

To characterise microstructural constituents in terms of composition, size, and morphology, additional *SEM*-based analysis methods are commonly used. These techniques play important roles in quantifying the effects of microstructure on damage and can also assist micromechanical analyses. In particular *EBSD*, which enables mapping of the crystallographic orientation of grains, plays a significant role in this work. When the primary electron beam interacts with a sample, incident electrons are scattered by atoms within the material. Depending on their scattering direction, these so-called backscattered electrons may then be diffracted by lattice planes within the crystal structure. If the Bragg condition is satisfied, constructive interference produces an amplified beam forming a cone-shaped pattern. The *EBSD* detector records this signal as projected patterns, known as "Kikuchi lines", which correspond to specific sets of lattice planes within the crystal. Because Kikuchi lines reflect the geometric arrangement of lattice planes, the crystallographic orientation at each point can be determined by analysing these patterns. [94] During *EBSD* analysis, the electron beam is scanned across the sample surface, mapping out crystallographic orientations at each scanned location. These maps enable determination of grain size and shape, serving for example as an essential preliminary tool for selecting appropriately oriented grains intended for subsequent micropillar compression experiments.

3.2.2 Measurement of constituents' mechanical properties

Analysing how microstructural constituents influence damage prevalence requires methods capable of measuring their individual mechanical properties, particularly for determining mechanical contrast. Micro- and nanomechanical testing methods such as micropillar compression and nanoindentation are widely used to obtain this information, as they can overcome macroscopic brittleness and the size restrictions posed by grain size and phase boundaries [95–98]. These methods enable measurement of individual grains or phases by using instrumented compression or hardness tests with tips on the micrometer scale. The resulting force-displacement curves are then analysed to extract relevant mechanical properties.

3.2.2.1 Nanoindentation

In a standard nanoindentation experiment, a three-sided self-similar pyramidal indenter known as a Berkovich tip, with a face half-angle of $\theta \approx 65.3^\circ$, is used to penetrate the specimen surface [95]. A sketch of this procedure is given in Figure 3.9 a).

For nanoindentation, a well-defined tip geometry is of particular importance, because the

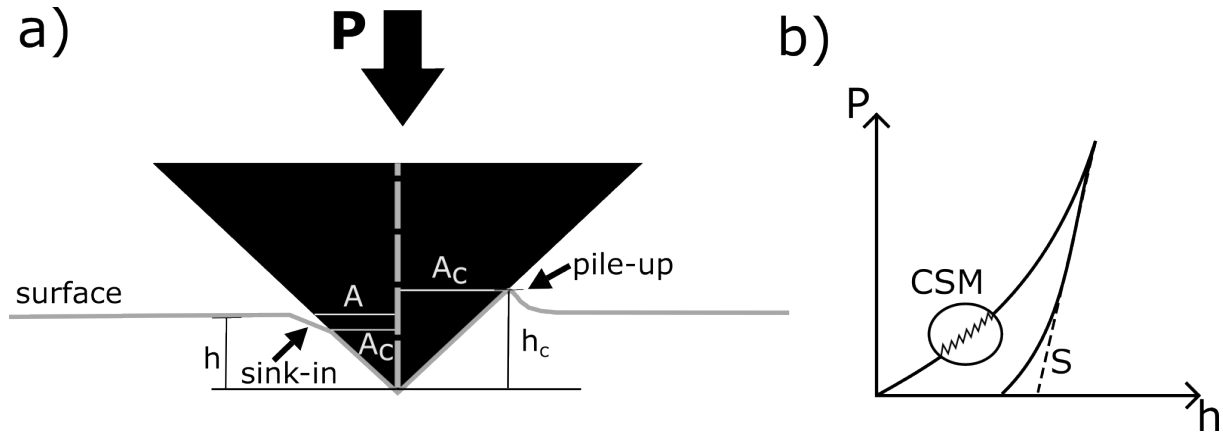


Figure 3.9: Sketch of typical nanoindentation procedure and related geometrical properties and effects in a). Sketch of a typical load over depth curve of a plastic material obtained via nanoindentation, with highlighted *CSM* oscillation and determination of unloading Stiffness *S* in b).

method relies on calculating the projected area A of the imprint at any depth h during the test by the means of a so called Diamond Area Function (*DAF*). For example, the *DAF* relating A and h_c for a Berkovich tip (cf. Figure 3.9 a)), which approximates a cone with the same depth to projected area ratio, is given by [95]:

$$A = 3\sqrt{3}h_c^2 \tan^2(\theta)$$

Here, h_c denotes the contact depth, which accounts for material effects like sink-in and pile-up. These effects can shift the reference point used to calculate the indentation depth away from the original sample surface. This is illustrated in Figure 3.9 a). The area determined by this method is then used to calculate mechanical properties such as hardness (H) and elastic modulus (E). The equation used to calculate the hardness then looks as follows:

$$H = \frac{P}{A}$$

The elastic modulus is determined from the unloading curve of a nanoindentation test as visualised in Figure 3.9 b). For this purpose, the unloading stiffness S , which is the initial shape of the unloading curve, is used in the following way [99]:

$$E = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A}}$$

During most load-controlled nanoindentation experiments, however, a controlled oscillation with relatively small amplitude is superimposed on a constant loading rate (cf. Figure 3.9 b)). This oscillation enables Continuous Stiffness Measurement (*CSM*), as each oscillation produces an unloading segment that provides values of hardness and elastic

modulus continuously throughout the indentation process. This approach achieves significant improvement over earlier methods where only hardness at maximum load/depth and elastic modulus from final unloading were evaluated [100]. In addition to hardness and elastic modulus, nanoindentation is capable of supplying more information on material behaviour when different indentation procedures are conducted. For example, it has been shown that strain-rate sensitivity can be measured via nanoindentation, enabling calculation of dislocation activation volume [101]. Additionally, complete stress-strain curves can be obtained by using spherical indentation tips and special calibration procedures [102]. Furthermore, recent advances in both instrumentation and testing protocols allowed reducing time between individual indents and employing shallower indentation depths, thus increasing both data collection rate and spatial resolution [103, 104]. Using these time savings, rapid nanoindentation mappings can be employed to profile the mechanical properties of complete microstructural areas. Usually, however, these mappings require additional preparation. In particular, to minimise indentation depth, the effect of the depth-dependent size effect must be determined beforehand, for example by regular nanoindentation. At shallower depths, steeper strain gradients necessitate formation of more geometrically necessary dislocations compared to deeper indentations, resulting in an apparent increase in measured hardness at low depths [105, 106]. The extent to which this effect is observed, depends on factors such as material type, processing history (e.g., forming/heat treatment), and even instrument calibration. Determining the minimum depth necessary to acquire a hardness value that is not effected by indentation depth, defines how far indents need to be spaced and how fast the test can be conducted. After mapping mechanical properties via rapid indent arrays, microstructural imaging can then reveal constituent locations and correlating position with measured property enables statistically robust analysis across multiphase microstructures.

3.2.2.2 Micropillar compression

Nanoindentation offers many benefits due to its high time efficiency and the broad applicability of the measured mechanical properties. However, there are certain material properties that cannot be determined using classical nanoindentation or rapid mapping techniques alone. This includes, for example, identifying a material's preferred slip system during deformation and its associated critical resolved shear stress. Although it is possible to observe individual slip events as surface steps around indents after nanoindentation and to correlate these with activated slip planes [107, 108], determining and interpreting the corresponding stresses remains challenging [109]. Insights on critical shear stresses on the same length scale as nanoindentation are more readily obtained using micropillar compression [97, 107]. The instrumentation for micropillar compression is similar in setup

to that used in nanoindentation but features several key differences. Instead of a sharp indenter tip, a flat punch is used. This allows compression of pillars with diameters up to several micrometers. Because accurate positioning is crucial, micropillar compression experiments are typically performed inside an electron microscope. This setup allows for direct visualisation of both the pillar and the flat punch tip, enabling precise alignment before and during compression. Because the flat punch has a flat surface and is not designed to penetrate the material, neither diamond area function calibration nor *CSM* protocols are required. An advantage, since *CSM* is known to influence measured mechanical properties [110]. Once tip and pillar are aligned, the pillar is deformed in approximately uniaxial compression and typically aborted before the pillars fails. The resulting load-depth data can be converted into stress-strain curves using known pillar dimensions. This enables analysis of hardening behaviour as well as extraction of yield points at plastic onset. Both features are known to be affected by size-effect [111], in particular the hardening behaviour is shown to vary strongly with pillar diameter [112] however without a clear material-overarching trend [113]. Nonetheless, identifying the onset point for plastic deformation remains especially valuable, because when combined with knowledge about crystallographic orientation via *EBSD* (i.e., Schmid factor calculation), this enables determination of critical resolved shear stress for specific slip systems. For this process, a pillar is usually analysed once more after deformation, to correlate slip steps on its surface with its crystallographic orientation and the spatial orientation of possible slip planes and directions [107]. The general workflow of identifying the critical resolved shear stress from a micropillar compression experiment is shown in Figure 3.10. Strength measurements at these scales not only facilitate calculation of constituent-level mechanical contrast [40], but also provide essential input parameters for multi-scale simulations, including those focused on damage evolution [115–117].

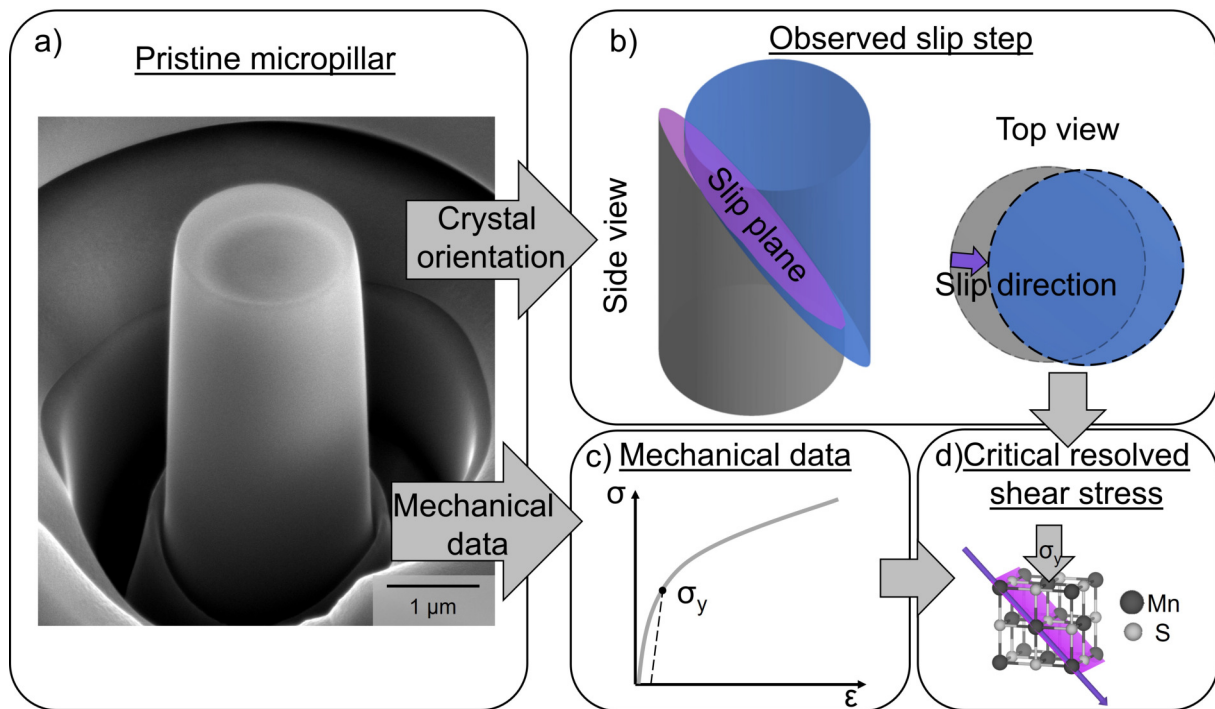


Figure 3.10: Workflow of determining the critical resolved shear stress of a slip system from a micropillar compression experiment. After the pristine micropillar shown in a) is compressed, the observed slip step is analysed to identify slip plane and slip direction as given in b). The correlated mechanical data shown in c) can be analysed to identify the point initial plastic deformation. Combining the identified slip system and the stress at onset of plastic deformation allows to identify the critical resolved shear stress for the observed slip system d). Crystal structure created with VESTA [114].

Chapter 4

Key insights and outlook

The following sections provide an overview of the main insights gained from this dissertation and explore potential further investigations.

4.1 Key insights

In this chapter the research questions addressed in Chapter 1 will be answered.

How can automated quantification of damage be achieved in steels exhibiting inclusion-induced damage?

Steels in which damage is primarily inclusion-induced differ significantly from those where damage arises mainly from inherent microstructural features, especially with respect to the geometric arrangement of their constituents. This distinction presents unique challenges for automated analysis. In particular, for the method introduced in subsubsection 3.2.1.1, this means it cannot be directly applied to datasets of microstructures different from the training data, without retraining. A key achievement of this dissertation is the development and successful demonstration of a novel two-step workflow for high-throughput automated quantification of inclusion-induced damage **Publication 1**. Because damage sites are localised within inclusions, it is sufficient first to identify these inclusions within the matrix (as demonstrated in **Publication 1** and visualised in Figure 4.1 a)).

Inclusions are larger than typical damage sites and exhibit consistent appearance with clear contrast to the surrounding matrix throughout the material, enabling efficient identification with minimal pre-analysis effort. In a second step, once inclusions are located, any associated damage sites can then be identified within them. This strategy offers several substantial advantages. Firstly, by enabling normalisation of measured damage parameters by inclusion count, it enhances comparability between samples, a crucial step towards establishing robust benchmarks across different materials or processing conditions. Secondly, because only easily distinguishable inclusions need to be considered, complex underlying microstructures do not complicate analysis. Notably, this removes the need for etching which can lead to artefacts in the microstructure and drastically alter the appearance depending on etching time and concentration of the etching agent. The methodology also exhibits versatility across different materials: as long as inclusion

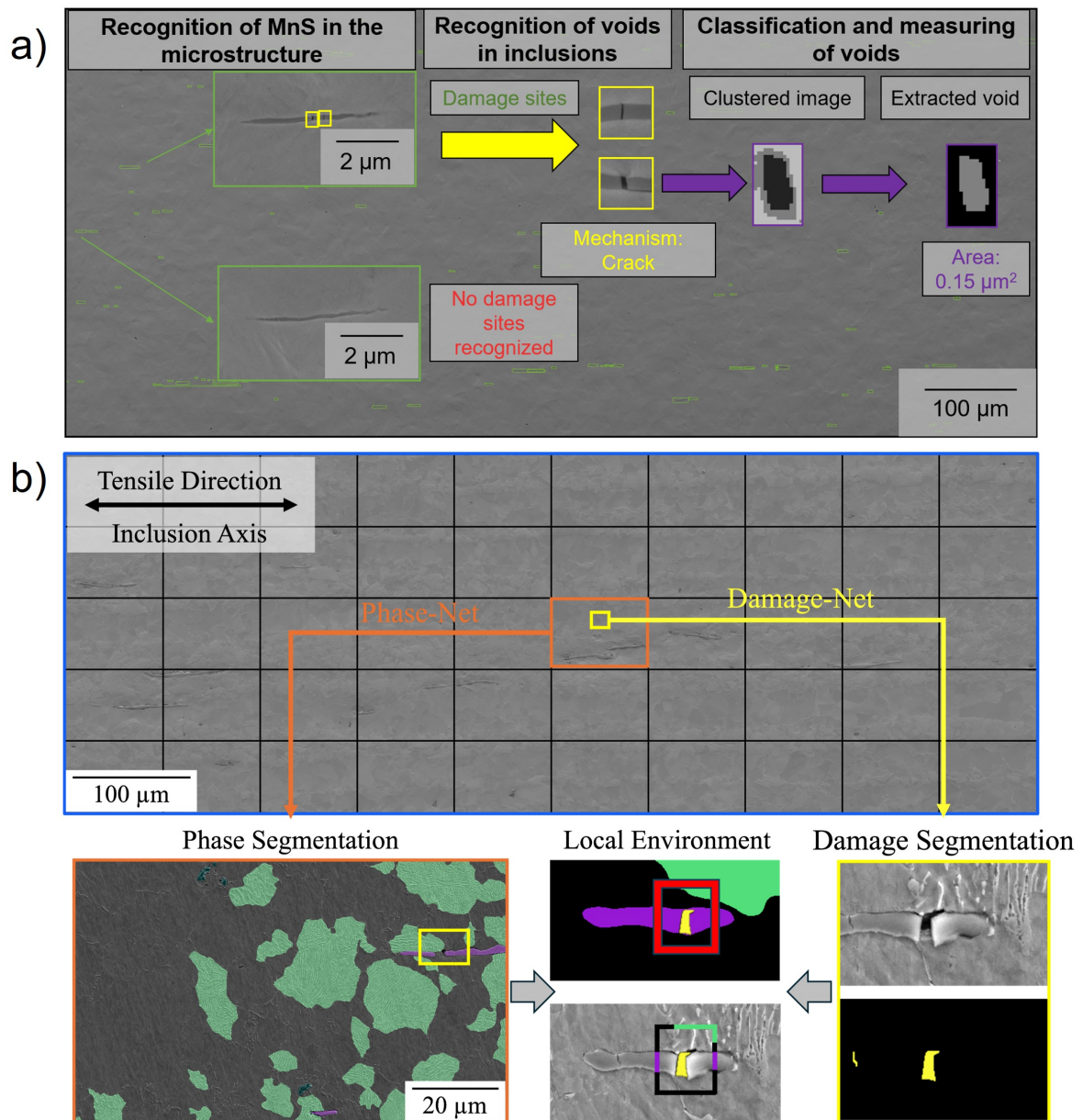


Figure 4.1: Workflow of quantifying damage prevalence by location of MnS and damage in unetched matrix in a) and segmentation of the etched microstructure in b). Reprinted from Publication 1 and Publication 4 respectively with the permission of Elsevier. [1]

morphology remains unchanged, it can be seamlessly applied across various heat-treated states or related alloys without further time-intensive training.

However, because the classification of the damage mechanisms is strongly dependent on the microstructure as seen for example in DP800 steels, these concepts remain applicable only to steels where the main damage mode is inclusion-induced. Additionally, while ignoring complex microstructural features simplifies damage analysis, it also provides less information compared to methods where the full microstructure is captured. In particular, local stress and strain distributions play a crucial role in damage nucleation and growth. These are influenced by all microstructural constituents, including those not visible using the approach described in **Publication 1**. To address these limitations we introduced a second novel method based on machine learning-driven image segmentation (**Publication 4**, see Figure 4.1 b). This method differs primarily by employing a machine learning network to segment an etched microstructure into its individual constituents, rather than simply locating inclusions within an unetched matrix as illustrated in Figure 4.1 b). The identified constituents include ferrite, pearlite, MnS inclusions, and damage sites. Since all constituent locations are known, the position and size of each damage site can be directly correlated with its neighbouring constituents. Although this method is less suitable for high-throughput determination of damage prevalence, because its applicability remains restricted to the specific 16MnCrS5 steel used to train the segmentation network, and results may be strongly influenced by *SEM* imaging conditions or metallographic preparation protocols, this approach represents a significant leap forward in fundamental research capability for damage prevalence in multi-phase materials.

How can strain path combinations and loading direction be used to strategically control damage prevalence through manipulation of stress state?

A major contribution of this dissertation is the demonstration that strain path combinations and loading direction changes can be strategically exploited to control damage prevalence by moving beyond global stress state parameters. Using the automated quantification workflow described above, damage prevalence in 16MnCrS5 samples was measured during an in-situ tensile test where the loading axis was aligned parallel to the longest dimension of *MnS* inclusions (**Publication 1**). The results show that under these conditions, fracture is the primary mode of damage initiation. Based on inspection of the resulting damage sites, we postulated that this mode would shift to delamination if the loading direction were rotated by 90° . This finding makes it clear that damage mechanisms depend on strain path, not solely on global stress state parameters like triaxiality or Lode parameter, but also due to inherent microstructural anisotropy. This insight provides a powerful tool for process engineers: by aligning loading directions with favourable microstructural orientations, it becomes possible to actively minimise damage formation during industrial forming operations. Notably, results from **Publication 2** demonstrate that this concept even extends to *DP* steel, which exhibits less pronounced anisotropy, when combining different strain paths, highlighting its applicability across different material classes. Specifically, when two forming steps with positive triaxiality are combined (such as bending followed by tension), reduced damage prevalence occurs if there is a 90° change in loading direction between steps. This effect was observed both for combinations of bending and tensile deformation in this work, as well as for two consecutive tensile tests reported in [118]. Combining bending and tensile tests also offers the benefit of enabling observation of regions where local triaxiality transitions between positive and negative values. The results highlighted two key observations: First, when damage sites are initiated during an initial forming step with positive triaxiality, they undergo shape change and volume reduction if a subsequent step with negative triaxiality is applied. Second, if the first forming step employs negative triaxiality, no damage sites are initiated, as expected. However, the damage prevalence after a subsequent positive triaxiality step is lower compared to material that did not experience prior forming with negative triaxiality, as illustrated in Figure 4.2. These findings establish clear design rules for forming processes: sequencing compressive before tensile deformation steps or rotating loading directions between steps can significantly reduce final damage levels.

The benefit of reduced damage prevalence can be attributed to strain hardening within ferrite, which reduces mechanical contrast between ferrite and martensite. Consequently, for DP800 steels, forming operations should maximise compressive stress states before applying processes involving high positive triaxialities.

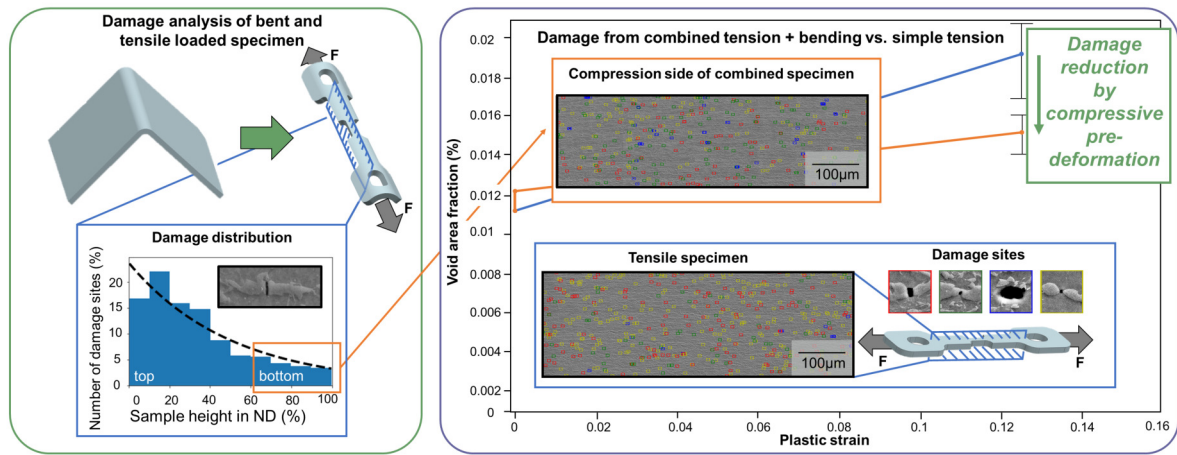


Figure 4.2: Reduction of damage prevalence in part of a bending and tensile test sample that experienced previous compressive stresses during forming. Reprinted from Publication 2 with the permission of Elsevier. [2]

How does mechanical contrast affect damage prevalence, and how can processing parameters be adjusted to exploit this effect?

The magnitude of mechanical contrast is a key factor influencing damage prevalence in 16MnCrS5 and we demonstrated in **Publication 3**, that it can be directly influenced by controlling the temperature. Tensile tests performed at temperatures ranging from ambient up to 600 °C revealed that damage prevalence does not vary linearly with temperature. This is due to the non-linear behaviour of mechanical contrast between *MnS* and the steel matrix as a function of temperature. Counter-intuitively, it was shown that damage prevalence can actually increase with temperature, for example, when going from 400 °C to 600 °C. Even though the yield strength of *MnS* becomes closer to that of the steel matrix when increasing the temperature from 400 °C to 600 °C, its hardening rate is actually higher at 600 °C. This results in increased damage prevalence and increased incompatibility of plastic deformation between *MnS* and the matrix during deformation, as determined by the difference in their strengths at a plastic strain of 5 %. Both trends are illustrated in Figure 4.3.

These results have immediate practical implications: forming processes should avoid temperatures such as 600 °C where increased mechanical contrast leads to higher damage prevalence. Instead, lower temperatures such as 400 °C, where reduced damage was observed, are preferable. Additionally, analysis revealed that certain orientations of *MnS* exhibit a yield strength anomaly, showing higher strength at 400 °C than at 200 °C. However, after considering all relevant factors (such as temperature, loading rate, stress state, polycrystallinity, and impurity concentrations), it was concluded that this anomaly arises only under very specific conditions and thus cannot be broadly incorporated into general models or estimations of mechanical contrast. Although this yield strength anomaly

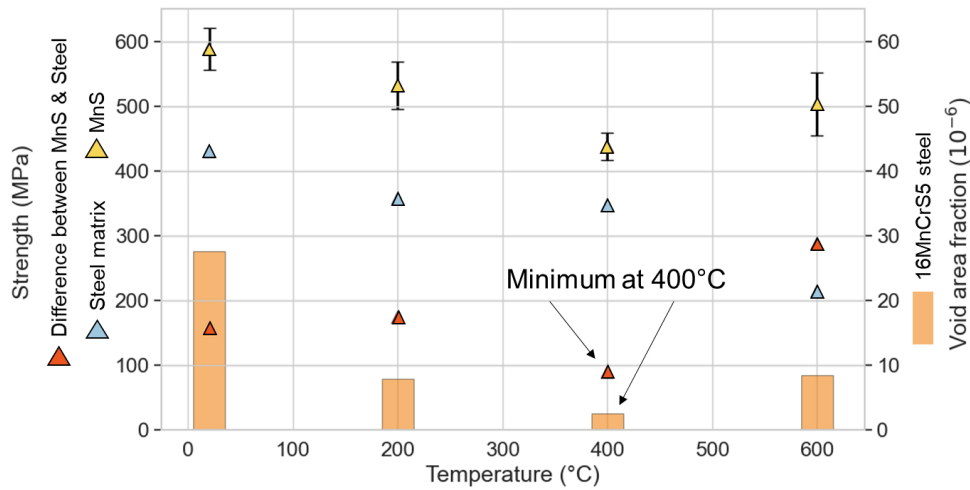


Figure 4.3: Trend of mechanical contrast and damage prevalence in terms of void area fraction for *MnS* and the steel matrix in 16MnCrS5 steel. Adapted from Publication 3 [3]. The original article was distributed under the terms of the Creative Commons CC-BY license.

may not affect general modelling, its existence demonstrates an additional possibility for targeted tuning of constituent properties, and therefore mechanical contrast. Finally, it should be noted that control of ferrite strength through strain hardening, achieved by tailoring strain paths as discussed in the previous section, constitutes an additional means of adjusting the mechanical contrast. This serves as yet another tool for designing mechanical contrast and further underlines the interconnectivity between the various strategies for achieving damage-controlled forming presented in this dissertation.

How do local phase environments affect damage prevalence in multi-phase microstructures?

A final opportunity presented in this thesis to control damage prevalence, with the goal of damage-controlled forming, lies in deliberate tailoring of microstructure. As described in subsubsection 3.1.3.2, certain microstructural designs, such as fine dispersion and uniform distribution of phases, have been shown to reduce damage prevalence in *DP* steels. However, it remains unclear whether these strategies can be directly applied to multi-phase materials such as 16MnCrS5. This uncertainty arises because the more complex network of phases and phase boundaries in multi-phase materials creates additional pathways for stress localisation, in particular around the damage-prone phase that are not present in simpler dual-phase systems. To address this gap, **Publication 4** analysed how local neighbouring constituents affect damage prevalence associated with *MnS* inclusions, in particular focussing on the nucleation and growth of damage sites as a function of their environment. These environments are: purely ferritic, purely pearlitic, and mixed environments. First, we focussed on determining the probability of damage site nucleation for each microstructural environment. Results showed that no clear trend emerged regarding

preferred locations of damage sites. This is in agreement with spherical nanoindentation measurements that we performed on the individual phases, showing that the yield strength of ferrite and pearlite is comparable. Consequently we concluded that damage nucleation, which is expected to occur at the onset of plastic deformation in MnS due to the low fracture strength of materials with rock-salt crystal structure [119, 120], is not strongly dependent on the local microstructural environment. Instead, other factors such as crystallographic orientation of matrix grains or inclusion morphology, resulting in local stress concentrations, are expected to have a greater impact. In a second step, we set out to determine the impact of the local environment on damage site growth by comparing the mean damage site size at various strain levels as a function of the pearlite fraction (Figure 4.4).

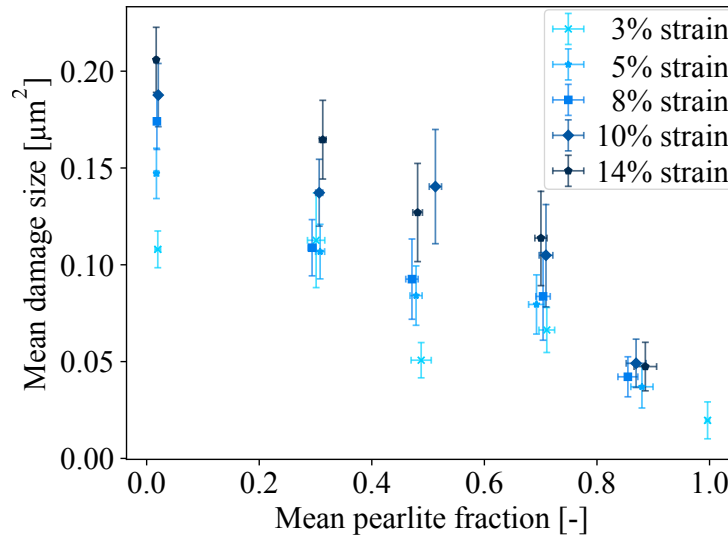


Figure 4.4: Trend of damage prevalence in terms of void area fraction in *MnS* as a function of the local environment of *MnS* inclusions. Adapted from Publication 4.

We chose this metric as any change in the mean damage site size can be explained either by growth of existing sites with increasing strain, which leads to an increase of the mean damage size, or by nucleation of smaller sites, which leads to a decrease. However, as stated previously, nucleation behaviour is not facilitated by any specific local environment. Thus, all observed trends in mean damage site size as a function of pearlite fraction are attributed solely to growth. Based on this reasoning, we showed that a ferritic environment facilitates damage site growth. From Figure 4.4, it is apparent that the strongest changes in mean size occur at high pearlite fractions: mean sizes are approximately halved when the pearlite fraction increases from around 70 % to 90 % at all strains. This indicates that damage sites almost completely engulfed in pearlite experience significantly less growth than those in other environments. We explain these observations based on the strain partitioning between ferrite and pearlite. In ferrite-pearlite microstructures most strain is accommodated by ferrite, due to its lower strain hardening capability compared

to pearlite, as apparent from our recorded spherical nanoindentation stress-strain curves. Consequently, pearlitic environments shield MnS interfaces from exposure to high strains, which mainly drives void growth. When comparing microstructural design principles from DP steels to the insights gained in **Publication 4**, it seems doubtful that DP design principles can be directly applied to multi-phase materials. Since small and finely distributed second phases are unlikely to lead to high phase fractions around damage-prone inclusions, it is expected that damage growth is increased in these microstructures.

4.2 Outlook and future work

In this dissertation, two tools for measuring damage prevalence were introduced, as well as three distinct strategies for modifying materials and processes with the aim of achieving damage-controlled forming. Based on these insights, further efforts are focussing on refining and implementing these concepts to enable practical damage-controlled forming. In the context of tools for damage analysis, one limitation is that measurements of damage prevalence are typically restricted to sample surfaces. However, since the microstructural environment affecting damage nucleation and growth forms a three-dimensional network around each inclusion, complete analysis cannot be achieved using only two-dimensional micrographs. Therefore, adapting the quantification approach presented in this thesis for use in slice-and-view experiments, such as those described in [59], could provide valuable insights into the three-dimensional nature of damage in steels dominated by inclusion-induced mechanisms. We expect this to be particularly helpful when combined with data from other methods, such as additional *EBSD* mappings and material models, gained for example from spherical nanoindentation. Together, these datasets enable the construction of three-dimensional representations of the microstructure, which can then be used to investigate local stress and strain distributions. This combinatory approach is planned for future work to shed light on which 3-dimensional microstructural arrangements are most important in the context of damage nucleation. Although several possible influences have been listed in **Publication 4**, the key factor driving damage nucleation in 16MnCrS5 remains to be revealed. An additional effect that has not been addressed is the superimposed influence of mechanical contrast on inclusion-induced damage prevalence. Because so far all work has focussed on spatial variation within existing microstructures rather than systematically altering phase properties, future work will involve engineering 16MnCrS5 steel microstructures. In this way, the previously determined influence of the microstructure on damage prevalence can be confirmed, while simultaneously elucidating the impact of changed mechanical properties of matrix constituents. To this end, three distinct microstructures will be compared: one featuring pearlitic bands surround-

ing MnS inclusions, a second with bainitic bands surrounding MnS inclusions, and a final microstructure comprising finely dispersed bainite. Based on this systematic variation, we expect to separate how mechanical contrast, based on the different strengths of pearlite and bainite, as well as microstructure, exemplified by a band like structure and a finely dispersed structure, each influence damage prevalence. Finally, future investigations will expand studies on 16MnCrS5 at elevated temperatures by examining the interaction of additional high-temperature processes, such as recrystallisation, and aspects of damage evolution, including nucleation, damage growth, and potentially even damage healing. The effects of recrystallisation on damage are still not fully understood, particularly on the scale of individual damage sites. Here, it remains an open question what effect changing the mechanical contrast and the local microstructure have on damage prevalence, in particular on damage site growth. Depending on heat-treatment conditions, we expect a range of effects to manifest, from softening of constituents, to grain sizes changes with corresponding effects on material strength, to development of preferred crystallographic orientations. Depending on which effect dominates the immediate environment of damage sites, we expect damage site growth to be influenced accordingly. Additionally, effects of larger scale, for example strain bands localizing deformation in certain areas are expected to form in partially recrystallized microstructures, leading to superimposed effects of the microstructural environment on damage site growth. For this purpose, the effects of complete and partial recrystallisation on both damage nucleation and subsequent growth will be evaluated by means of stopped tensile tests and in-situ heat-treatment. By revealing the interactions between (partial) recrystallisation and damage, we hope to provide deeper understanding of the interaction of heat-treatment and damage.

Bibliography

- [1] M. A. Wollenweber, C. F. Kusche, T. Al-Samman, and S. Korte-Kerzel, “On the automated characterisation of inclusion-induced damage in 16mncrs5 case-hardening steel,” *Advances in Industrial and Manufacturing Engineering*, vol. 7, p. 100123, 2023.
- [2] M. A. Wollenweber, S. Medghalchi, L. R. Guimarães, N. Lohrey, C. F. Kusche, U. Kerzel, T. Al-Samman, and S. Korte-Kerzel, “On the damage behaviour in dual-phase dp800 steel deformed in single and combined strain paths,” *Materials & Design*, vol. 231, p. 112016, 2023.
- [3] M. A. Wollenweber, J. Werner, C. F. Kusche, C. Tian, P.-L. Sun, J. Gerlach, T. Al-Samman, and S. Korte-Kerzel, “On the plasticity of mns at elevated temperatures and its influence on damage prevalence,” *Acta Materialia*, vol. 291, p. 120987, 2025.
- [4] M. A. Wollenweber, T. Reclik, C. Reinhart, V. Maier-Kiener, T. Al-Samman, and S. Korte-Kerzel, “Ductile damage at mns inclusions in 16mncrs5 steel - influence of the phase environment,” *RWTH Publications preprint RWTH-2025-10405*, 2025.
- [5] UNEP, *Global resources outlook 2019: Natural resources for the future we want*. UN, 2020.
- [6] S. H. Farjana, N. Huda, M. P. Mahmud, and R. Saidur, “A review on the impact of mining and mineral processing industries through life cycle assessment,” *Journal of Cleaner Production*, vol. 231, pp. 1200–1217, 2019.
- [7] M. J. de Villafranca Casas, S. Smit, A. Nilsson, and T. Kuramochi, “Climate targets by major steel companies: an assessment of collective ambition and planned emission reduction measures,” *Energy and Climate Change*, vol. 5, p. 100120, 2024.
- [8] I. Fărcean, G. Proștean, and A. Socalici, “Sustainable development indicators in the steel industry,” in *Journal of Physics: Conference Series*, vol. 2540, p. 012045, IOP Publishing, 2023.
- [9] W. S. Association *et al.*, “Climate change and the production of iron and steel,” *World Steel Association: Brussels, Belgium*, 2021.
- [10] Y. Ma, I. R. Souza Filho, Y. Bai, J. Schenk, F. Patisson, A. Beck, J. A. van Bokhoven, M. G. Willinger, K. Li, D. Xie, *et al.*, “Hierarchical nature of hydrogen-based direct reduction of iron oxides,” *Scripta Materialia*, vol. 213, p. 114571, 2022.

- [11] K. Rechberger, A. Spanlang, A. Sasiain Conde, H. Wolfmeir, and C. Harris, “Green hydrogen-based direct reduction for low-carbon steelmaking,” *steel research international*, vol. 91, no. 11, p. 2000110, 2020.
- [12] G. Zang, P. Sun, A. Elgowainy, P. Bobba, C. McMillan, O. Ma, K. Podkaminer, N. Rustagi, M. Melaina, and M. Koleva, “Cost and life cycle analysis for deep co2 emissions reduction of steelmaking: Blast furnace-basic oxygen furnace and electric arc furnace technologies,” *International Journal of Greenhouse Gas Control*, vol. 128, p. 103958, 2023.
- [13] Y. Zhang, L. Yu, K. Cui, H. Wang, and T. Fu, “Carbon capture and storage technology by steel-making slags: Recent progress and future challenges,” *Chemical Engineering Journal*, vol. 455, p. 140552, 2023.
- [14] T. S. Cao, “Models for ductile damage and fracture prediction in cold bulk metal forming processes: a review,” *International Journal of Material Forming*, vol. 10, no. 2, pp. 139–171, 2017.
- [15] A. Tekkaya, P.-O. Bouchard, S. Bruschi, and C. Tasan, “Damage in metal forming,” *CIRP Annals*, vol. 69, no. 2, pp. 600–623, 2020.
- [16] K. Hectors and W. De Waele, “Cumulative damage and life prediction models for high-cycle fatigue of metals: A review,” *Metals*, vol. 11, no. 2, p. 204, 2021.
- [17] J. A. Stendal, M. Eisentraut, M. Imran, I. Sizova, S. Bolz, S. Weiß, and M. Bambach, “Accelerated hot deformation and heat treatment of the tial alloy tnm-b1 for enhanced hot workability and controlled damage,” *Journal of Materials Processing Technology*, vol. 291, p. 116999, 2021.
- [18] R. Gitschel, O. Hering, A. Schulze, and A. Erman Tekkaya, “Controlling damage evolution in geometrically identical cold forged parts by counterpressure,” *Journal of Manufacturing Science and Engineering*, vol. 145, no. 1, p. 011011, 2023.
- [19] P. Ludwik, “Bestimmung der reißfestigkeit aus der gleichmäßigen dehnung,” *International Journal of Materials Research*, vol. 18, no. 1-12, pp. 269–272, 1926.
- [20] W. Remmers, “Causes of cuppy wire,” *Trans Metall Soc AIME*, vol. 89, pp. 107–120, 1930.
- [21] A. Weck, D. Wilkinson, E. Maire, and H. Toda, “Visualization by x-ray tomography of void growth and coalescence leading to fracture in model materials,” *Acta Materialia*, vol. 56, no. 12, pp. 2919–2928, 2008.

- [22] J. Besson, “Continuum models of ductile fracture: a review,” *International Journal of Damage Mechanics*, vol. 19, no. 1, pp. 3–52, 2010.
- [23] C. C. Tasan, M. Diehl, D. Yan, M. Bechtold, F. Roters, L. Schemmann, C. Zheng, N. Peranio, D. Ponge, M. Koyama, *et al.*, “An overview of dual-phase steels: advances in microstructure-oriented processing and micromechanically guided design,” *Annual Review of Materials Research*, vol. 45, no. 1, pp. 391–431, 2015.
- [24] D. Yan, C. C. Tasan, and D. Raabe, “High resolution in situ mapping of microstrain and microstructure evolution reveals damage resistance criteria in dual phase steels,” *Acta Materialia*, vol. 96, pp. 399–409, 2015.
- [25] G. Avramovic-Cingara, Y. Ososkov, M. Jain, and D. Wilkinson, “Effect of martensite distribution on damage behaviour in dp600 dual phase steels,” *Materials Science and Engineering: A*, vol. 516, no. 1-2, pp. 7–16, 2009.
- [26] T. F. Morgeneyer, M. Starink, and I. Sinclair, “Evolution of voids during ductile crack propagation in an aluminium alloy sheet toughness test studied by synchrotron radiation computed tomography,” *Acta Materialia*, vol. 56, no. 8, pp. 1671–1679, 2008.
- [27] E. Maire, S. Zhou, J. Adrien, and M. Dimichiel, “Damage quantification in aluminium alloys using in situ tensile tests in x-ray tomography,” *Engineering Fracture Mechanics*, vol. 78, no. 15, pp. 2679–2690, 2011.
- [28] F. Hannard, T. Pardoen, É. Maire, C. Le Boulrot, R. Mokso, and A. Simar, “Characterization and micromechanical modelling of microstructural heterogeneity effects on ductile fracture of 6xxx aluminium alloys,” *Acta Materialia*, vol. 103, pp. 558–572, 2016.
- [29] R. Meya, C. F. Kusche, C. Löbke, T. Al-Samman, S. Korte-Kerzel, and A. E. Tekkaya, “Global and high-resolution damage quantification in dual-phase steel bending samples with varying stress states,” *Metals*, vol. 9, no. 3, p. 319, 2019.
- [30] F. Guhr, R. Gitschel, F.-J. Barthold, and A. E. Tekkaya, “Numerical optimisation of damage in extrusion processes,” *PAMM*, vol. 23, no. 3, p. e202300199, 2023.
- [31] N. Fehlemann, M. HENRICH, M. Müller, M. KÖNEMANN, T. Bergs, and S. Münstermann, “Fatigue resistance of deep drawn parts: A scale bridging simulative study using representative volume elements and crystal plasticity simulations,” *Materials Research Proceedings*, vol. 41, 2024.

- [32] D. Güzel, T. Kaiser, and A. Menzel, “A computational multiscale approach towards the modelling of microstructures with material interfaces in electrical conductors,” *Mathematics and Mechanics of Solids*, vol. 30, no. 2, pp. 247–266, 2025.
- [33] T. Clausmeyer, A. Schowtjak, S. Wang, R. Gitschel, O. Hering, P. Pavliuchenko, J. Lohmar, R. Ostwald, G. Hirt, and A. E. Tekkaya, “Prediction of ductile damage in the process chain of caliber rolling and forward rod extrusion,” *Procedia Manufacturing*, vol. 47, pp. 649–655, 2020.
- [34] M. Müller, I. F. Weiser, T. Herrig, and T. Bergs, “Numerical prediction of the influence of process parameters and process set-up on damage evolution during deep drawing of rectangular cups,” *Engineering Proceedings*, vol. 26, no. 1, p. 6, 2022.
- [35] O. Hering, A. Dunlap, A. E. Tekkaya, A. Aretz, and A. Schwedt, “Characterization of damage in forward rod extruded parts,” *International Journal of Material Forming*, vol. 13, pp. 1003–1014, 2020.
- [36] C. Kusche, T. Reclik, M. Freund, T. Al-Samman, U. Kerzel, and S. Korte-Kerzel, “Large-area, high-resolution characterisation and classification of damage mechanisms in dual-phase steel using deep learning,” *PloS one*, vol. 14, no. 5, p. e0216493, 2019.
- [37] C. F. Kusche, F. Pütz, S. Münstermann, T. Al-Samman, and S. Korte-Kerzel, “On the effect of strain and triaxiality on void evolution in a heterogeneous microstructure—a statistical and single void study of damage in dp800 steel,” *Materials Science and Engineering: A*, vol. 799, p. 140332, 2021.
- [38] R. Lapovok and D. Hodgson, “A damage accumulation model for complex strain paths: Prediction of ductile failure in metals,” *Journal of the Mechanics and Physics of Solids*, vol. 57, no. 11, pp. 1851–1864, 2009.
- [39] F. Pütz, F. Shen, M. Könemann, and S. Münstermann, “The differences of damage initiation and accumulation of dp steels: a numerical and experimental analysis,” *International Journal of Fracture*, vol. 226, pp. 1–15, 2020.
- [40] C. Tian, D. Ponge, L. Christiansen, and C. Kirchlechner, “On the mechanical heterogeneity in dual phase steel grades: Activation of slip systems and deformation of martensite in dp800,” *Acta Materialia*, vol. 183, pp. 274–284, 2020.

- [41] J. P. Hoefnagels, C. C. Tasan, F. Maresca, F. J. Peters, and V. G. Kouznetsova, “Retardation of plastic instability via damage-enabled microstrain delocalization,” *Journal of materials science*, vol. 50, pp. 6882–6897, 2015.
- [42] X. Li, S. Wang, J. Lohmar, and G. Hirt, “Design of caliber rolls incorporating load path dependent damage evolution,” *Procedia manufacturing*, vol. 47, pp. 643–648, 2020.
- [43] J. Lemaitre and J. Dufailly, “Damage measurements,” *Engineering Fracture Mechanics*, vol. 28, no. 5-6, pp. 643–661, 1987.
- [44] Z. Shi, H. Ma, and J. Li, “A novel damage variable to characterize evolution of microstructure with plastic deformation for ductile metal materials under tensile loading,” *Engineering Fracture Mechanics*, vol. 78, no. 3, pp. 503–513, 2011.
- [45] B. Kiefer, T. Waffenschmidt, L. Sprave, and A. Menzel, “A gradient-enhanced damage model coupled to plasticity—multi-surface formulation and algorithmic concepts,” *International Journal of Damage Mechanics*, vol. 27, no. 2, pp. 253–295, 2018.
- [46] L. Sprave and A. Menzel, “A large strain anisotropic ductile damage model—effective driving forces and gradient-enhancement of damage vs. plasticity,” *Computer Methods in Applied Mechanics and Engineering*, vol. 416, p. 116284, 2023.
- [47] C. Tasan, J. Hoefnagels, and M. Geers, “Identification of the continuum damage parameter: An experimental challenge in modeling damage evolution,” *Acta Materialia*, vol. 60, no. 8, pp. 3581–3589, 2012.
- [48] G. Hirt, A. E. Tekkaya, T. Clausmeyer, and J. Lohmar, “Potential and status of damage controlled forming processes,” 2020.
- [49] D. Hull and D. Rimmer, “The growth of grain-boundary voids under stress,” *Philosophical Magazine*, vol. 4, no. 42, pp. 673–687, 1959.
- [50] L. Liu, F. Maresca, T. Vermeij, J. P. Hoefnagels, M. G. Geers, and V. G. Kouznetsova, “An integrated experimental-numerical study of martensite/ferrite interface damage initiation in dual-phase steels,” *Scripta Materialia*, vol. 239, p. 115798, 2024.
- [51] K. Gall, N. Yang, H. Sehitoglu, and Y. I. Chumlyakov, “Fracture of precipitated niti shape memory alloys,” *International journal of fracture*, vol. 109, pp. 189–207, 2001.

- [52] K. Alharbi, H. Ghadbeigi, P. Efthymiadis, M. Zanganeh, S. Celotto, R. Dashwood, and C. Pinna, “Damage in dual phase steel dp1000 investigated using digital image correlation and microstructure simulation,” *Modelling and Simulation in Materials Science and Engineering*, vol. 23, no. 8, p. 085005, 2015.
- [53] R. Becker, A. Needleman, S. Suresh, V. Tvergaard, and A. Vasudevan, “An analysis of ductile failure by grain boundary void growth,” *Acta Metallurgica*, vol. 37, no. 1, pp. 99–120, 1989.
- [54] L. A. Lingnau, J. Heermant, J. L. Otto, K. Donnerbauer, L. M. Sauer, L. Lücker, M. Macias Barrientos, and F. Walther, “Separation of damage mechanisms in full forward rod extruded case-hardening steel 16mncrs5 using 3d image segmentation,” *Materials*, vol. 17, no. 12, p. 3023, 2024.
- [55] C. Tian and C. Kirchlechner, “The fracture toughness of martensite islands in dual-phase dp800 steel,” *Journal of Materials Research*, vol. 36, pp. 2495–2504, 2021.
- [56] M. Calcagnotto, Y. Adachi, D. Ponge, and D. Raabe, “Deformation and fracture mechanisms in fine-and ultrafine-grained ferrite/martensite dual-phase steels and the effect of aging,” *Acta Materialia*, vol. 59, no. 2, pp. 658–670, 2011.
- [57] K. Park, M. Nishiyama, N. Nakada, T. Tsuchiyama, and S. Takaki, “Effect of the martensite distribution on the strain hardening and ductile fracture behaviors in dual-phase steel,” *Materials Science and Engineering: A*, vol. 604, pp. 135–141, 2014.
- [58] J. Zhang, H. Di, Y. Deng, and R. Misra, “Effect of martensite morphology and volume fraction on strain hardening and fracture behavior of martensite–ferrite dual phase steel,” *Materials Science and Engineering: A*, vol. 627, pp. 230–240, 2015.
- [59] S. Medghalchi, E. Karimi, S.-H. Lee, B. Berkels, U. Kerzel, and S. Korte-Kerzel, “Three-dimensional characterisation of deformation-induced damage in dual phase steel using deep learning,” *Materials & Design*, vol. 232, p. 112108, 2023.
- [60] F. Qayyum, M. Umar, V. Elagin, M. Kirschner, F. Hoffmann, S. Guk, and U. Prahl, “Influence of non-metallic inclusions on local deformation and damage behavior of modified 16mncrs5 steel,” *Crystals*, vol. 12, no. 2, p. 281, 2022.
- [61] P. J. Noell, J. D. Carroll, and B. L. Boyce, “The mechanisms of ductile rupture,” *Acta Materialia*, vol. 161, pp. 83–98, 2018.
- [62] W. Wciślik and S. Lipiec, “Void-induced ductile fracture of metals: experimental observations,” *Materials*, vol. 15, no. 18, p. 6473, 2022.

- [63] R. Thomson and J. Hancock, "Ductile failure by void nucleation, growth and coalescence," *International Journal of Fracture*, vol. 26, pp. 99–112, 1984.
- [64] G. Avramovic-Cingara, C. A. Saleh, M. Jain, and D. Wilkinson, "Void nucleation and growth in dual-phase steel 600 during uniaxial tensile testing," *Metallurgical and materials transactions A*, vol. 40, pp. 3117–3127, 2009.
- [65] A. A. Benzerga and J.-B. Leblond, "Ductile fracture by void growth to coalescence," *Advances in applied mechanics*, vol. 44, pp. 169–305, 2010.
- [66] J. R. Rice and D. M. Tracey, "On the ductile enlargement of voids in triaxial stress fields," *Journal of the Mechanics and Physics of Solids*, vol. 17, no. 3, pp. 201–217, 1969.
- [67] J. Kim, X. Gao, and T. S. Srivatsan, "Modeling of void growth in ductile solids: effects of stress triaxiality and initial porosity," *Engineering fracture mechanics*, vol. 71, no. 3, pp. 379–400, 2004.
- [68] N. Bonora, D. Gentile, A. Pirondi, and G. Newaz, "Ductile damage evolution under triaxial state of stress: theory and experiments," *International Journal of Plasticity*, vol. 21, no. 5, pp. 981–1007, 2005.
- [69] G. Gottstein, *Physical foundations of materials science*, vol. 3. Springer, 2004.
- [70] R. Gitschel, J. Gebhard, Y. P. Korkolis, and A. E. Tekkaya, "Isolating the effects of deviatoric and hydrostatic stress on damage evolution using cold extrusion experiments," *International Journal of Solids and Structures*, vol. 310, p. 113205, 2025.
- [71] Y. Bao and T. Wierzbicki, "On the cut-off value of negative triaxiality for fracture," *Engineering fracture mechanics*, vol. 72, no. 7, pp. 1049–1069, 2005.
- [72] K. Zhang, J. Bai, and D. Francois, "Numerical analysis of the influence of the lode parameter on void growth," *International Journal of Solids and Structures*, vol. 38, no. 32-33, pp. 5847–5856, 2001.
- [73] H. R. Kotadia, G. Gibbons, A. Das, and P. D. Howes, "A review of laser powder bed fusion additive manufacturing of aluminium alloys: Microstructure and properties," *Additive Manufacturing*, vol. 46, p. 102155, 2021.
- [74] R. B. Figueiredo, M. Kawasaki, and T. G. Langdon, "Seventy years of hall-petch, ninety years of superplasticity and a generalized approach to the effect of grain size on flow stress," *Progress in Materials Science*, vol. 137, p. 101131, 2023.

- [75] L. Berners, P.-L. Sun, A. Mikitisin, J. Mayer, and S. Korte-Kerzel, “Influence of microstructure refinement on co-plasticity in an mg-4.7 al-2.9 ca alloy,” *Journal of Alloys and Compounds*, p. 180516, 2025.
- [76] C. C. Tasan, J. P. Hoefnagels, M. Diehl, D. Yan, F. Roters, and D. Raabe, “Strain localization and damage in dual phase steels investigated by coupled in-situ deformation experiments and crystal plasticity simulations,” *International Journal of Plasticity*, vol. 63, pp. 198–210, 2014.
- [77] F. Pütz, N. Fehlemann, V. Göksu, M. Henrich, M. Könemann, and S. Münstermann, “A data driven computational microstructure analysis on the influence of martensite banding on damage in dp-steels,” *Computational Materials Science*, vol. 218, p. 111903, 2023.
- [78] T. Koseki, J. Inoue, and S. Nambu, “Development of multilayer steels for improved combinations of high strength and high ductility,” *Materials transactions*, vol. 55, no. 2, pp. 227–237, 2014.
- [79] M. Azuma, S. Goutianos, N. Hansen, G. Winther, and X. Huang, “Effect of hardness of martensite and ferrite on void formation in dual phase steel,” *Materials Science and Technology*, vol. 28, no. 9-10, pp. 1092–1100, 2012.
- [80] H. Ghassemi-Armaki, R. Maaß, S. Bhat, S. Sriram, J. Greer, and K. Kumar, “Deformation response of ferrite and martensite in a dual-phase steel,” *Acta Materialia*, vol. 62, pp. 197–211, 2014.
- [81] Y. Mazaheri, A. Kermanpur, and A. Najafizadeh, “Nanoindentation study of ferrite–martensite dual phase steels developed by a new thermomechanical processing,” *Materials Science and Engineering: A*, vol. 639, pp. 8–14, 2015.
- [82] K. Hasegawa, K. Kawamura, T. Urabe, and Y. Hosoya, “Effects of microstructure on stretch-flange-formability of 980 mpa grade cold-rolled ultra high strength steel sheets,” *ISIJ international*, vol. 44, no. 3, pp. 603–609, 2004.
- [83] X. Sun, K. S. Choi, W. N. Liu, and M. A. Khaleel, “Predicting failure modes and ductility of dual phase steels using plastic strain localization,” *International Journal of Plasticity*, vol. 25, no. 10, pp. 1888–1909, 2009.
- [84] W. Woo, V. Em, E.-Y. Kim, S. Han, Y. Han, and S.-H. Choi, “Stress–strain relationship between ferrite and martensite in a dual-phase steel studied by in situ neutron diffraction and crystal plasticity theories,” *Acta materialia*, vol. 60, no. 20, pp. 6972–6981, 2012.

- [85] M. D. Taylor, K. S. Choi, X. Sun, D. K. Matlock, C. Packard, L. Xu, and F. Barlat, “Correlations between nanoindentation hardness and macroscopic mechanical properties in dp980 steels,” *Materials Science and Engineering: A*, vol. 597, pp. 431–439, 2014.
- [86] C. Scott, B. S. Amirkhiz, I. Pushkareva, F. Fazeli, S. Allain, and H. Azizi, “New insights into martensite strength and the damage behaviour of dual phase steels,” *Acta Materialia*, vol. 159, pp. 112–122, 2018.
- [87] C. Tian, C. F. Kusche, A. Medina, S. Lee, M. A. Wollenweber, R. Pippan, S. Korte-Kerzel, and C. Kirchlechner, “Understanding the damage initiation and growth mechanisms of two dp800 dual phase grades,” *Materials & Design*, vol. 238, p. 112630, 2024.
- [88] H. Chao, L. H. Van Vlack, F. Oberlin, and L. Thomassen, “Inclusion deformation: I: The mechanism of fracture and deformation in mns.,” tech. rep., Department of Chemical and Metallurgical Engineering, The University of Michigan, 1962.
- [89] S. Furuseth and A. Kjekshus, “On the properties of a-mns and mns₂,” *Acta Chem. Scand*, vol. 19, no. 6, pp. 1405–1410, 1965.
- [90] C. F. Kusche, J. S.-L. Gibson, M. A. Wollenweber, and S. Korte-Kerzel, “On the mechanical properties and deformation mechanisms of manganese sulphide inclusions,” *Materials & Design*, vol. 193, p. 108801, 2020.
- [91] S. Medghalchi, J. Kortmann, S.-H. Lee, E. Karimi, U. Kerzel, and S. Korte-Kerzel, “Automated segmentation of large image datasets using artificial intelligence for microstructure characterisation and damage analysis,” *Materials & Design*, vol. 243, p. 113031, 2024.
- [92] S. Medghalchi, C. F. Kusche, E. Karimi, U. Kerzel, and S. Korte-Kerzel, “Damage analysis in dual-phase steel using deep learning: transfer from uniaxial to biaxial straining conditions by image data augmentation,” *Jom*, vol. 72, pp. 4420–4430, 2020.
- [93] T. Reclik, S. Medghalchi, P. Schumacher, M. Wollenweber, T. Al-Samman, S. Korte-Kerzel, and U. Kerzel, “Resolution enhancement of scanning electron micrographs using artificial intelligence,” *Materials & Design*, vol. 253, p. 113955, 2025.
- [94] A. J. Schwartz, M. Kumar, B. L. Adams, and D. P. Field, *Electron backscatter diffraction in materials science*, vol. 2. Springer, 2009.

- [95] A. C. Fischer-Cripps, “Critical review of analysis and interpretation of nanoindentation test data,” *Surface and coatings technology*, vol. 200, no. 14-15, pp. 4153–4165, 2006.
- [96] C. A. Schuh, “Nanoindentation studies of materials,” *Materials today*, vol. 9, no. 5, pp. 32–40, 2006.
- [97] M. D. Uchic, P. A. Shade, and D. M. Dimiduk, “Plasticity of micrometer-scale single crystals in compression,” *Annual Review of Materials Research*, vol. 39, no. 1, pp. 361–386, 2009.
- [98] S. Korte and W. Clegg, “Micropillar compression of ceramics at elevated temperatures,” *Scripta Materialia*, vol. 60, no. 9, pp. 807–810, 2009.
- [99] W. C. Oliver and G. M. Pharr, “An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments,” *Journal of materials research*, vol. 7, no. 6, pp. 1564–1583, 1992.
- [100] W. C. Oliver and G. M. Pharr, “Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology,” *Journal of materials research*, vol. 19, no. 1, pp. 3–20, 2004.
- [101] V. Maier-Kiener and K. Durst, “Advanced nanoindentation testing for studying strain-rate sensitivity and activation volume,” *Jom*, vol. 69, pp. 2246–2255, 2017.
- [102] A. Leitner, V. Maier-Kiener, and D. Kiener, “Essential refinements of spherical nanoindentation protocols for the reliable determination of mechanical flow curves,” *Materials & Design*, vol. 146, pp. 69–80, 2018.
- [103] M. Sebastiani, R. Moscatelli, F. Ridi, P. Baglioni, and F. Carassiti, “High-resolution high-speed nanoindentation mapping of cement pastes: Unravelling the effect of microstructure on the mechanical properties of hydrated phases,” *Materials & Design*, vol. 97, pp. 372–380, 2016.
- [104] J. M. Wheeler, “Mechanical phase mapping of the taza meteorite using correlated high-speed nanoindentation and edx,” *Journal of Materials Research*, vol. 36, pp. 94–104, 2021.
- [105] W. D. Nix and H. Gao, “Indentation size effects in crystalline materials: a law for strain gradient plasticity,” *Journal of the Mechanics and Physics of Solids*, vol. 46, no. 3, pp. 411–425, 1998.

- [106] Y. Huang, F. Zhang, K. Hwang, W. Nix, G. Pharr, and G. Feng, “A model of size effects in nano-indentation,” *Journal of the Mechanics and Physics of Solids*, vol. 54, no. 8, pp. 1668–1686, 2006.
- [107] J. S.-L. Gibson, R. Pei, M. Heller, S. Medghalchi, W. Luo, and S. Korte-Kerzel, “Finding and characterising active slip systems: a short review and tutorial with automation tools,” *Materials*, vol. 14, no. 2, p. 407, 2021.
- [108] M. Freund, Z. Xie, P.-L. Sun, L. Berners, J. Spille, H. Wang, C. Thomas, M. Feuerbacher, M. Lipinska-Chwalek, J. Mayer, *et al.*, “Influence of chemical composition on the room temperature plasticity of c15 ca-al-mg laves phases,” *Acta Materialia*, vol. 276, p. 120124, 2024.
- [109] W. Gerberich, J. Nelson, E. Lilleodden, P. Anderson, and J. Wyrobek, “Indentation induced dislocation nucleation: The initial yield point,” *Acta Materialia*, vol. 44, no. 9, pp. 3585–3598, 1996.
- [110] G. M. Pharr, J. H. Strader, and W. Oliver, “Critical issues in making small-depth mechanical property measurements by nanoindentation with continuous stiffness measurement,” *Journal of Materials Research*, vol. 24, no. 3, pp. 653–666, 2009.
- [111] R. Soler, J. M. Wheeler, H.-J. Chang, J. Segurado, J. Michler, J. Llorca, and J. M. Molina-Aldareguia, “Understanding size effects on the strength of single crystals through high-temperature micropillar compression,” *Acta materialia*, vol. 81, pp. 50–57, 2014.
- [112] D. Kiener, P. Guruprasad, S. M. Keralavarma, G. Dehm, and A. A. Benzerga, “Work hardening in micropillar compression: In situ experiments and modeling,” *Acta Materialia*, vol. 59, no. 10, pp. 3825–3840, 2011.
- [113] C. Frick, B. Clark, S. Orso, A. Schneider, and E. Arzt, “Size effect on strength and strain hardening of small-scale [1 1 1] nickel compression pillars,” *Materials Science and Engineering: A*, vol. 489, no. 1-2, pp. 319–329, 2008.
- [114] K. Momma and F. Izumi, “Vesta 3 for three-dimensional visualization of crystal, volumetric and morphology data,” *Applied Crystallography*, vol. 44, no. 6, pp. 1272–1276, 2011.
- [115] A. Cruzado, B. Gan, M. Jiménez, D. Barba, K. Ostolaza, A. Linaza, J. Molina-Aldareguia, J. Llorca, and J. Segurado, “Multiscale modeling of the mechanical behavior of in718 superalloy based on micropillar compression and computational homogenization,” *Acta Materialia*, vol. 98, pp. 242–253, 2015.

- [116] F.-J. Gallardo-Basile, F. Roters, R. M. Jentner, J. P. Best, C. Kirchlechner, K. Srivastava, S. Scholl, and M. Diehl, “Application of a nanoindentation-based approach for parameter identification to a crystal plasticity model for bcc metals,” *Materials Science and Engineering: A*, vol. 881, p. 145373, 2023.
- [117] N. C. Fehlemann, A. Medina, S. Lee, C. Kirchlechner, and S. Münstermann, “Crystal plasticity parameter identification via statistical relevant micropillar compression,” *Acta Materialia*, vol. 297, p. 121321, 2025.
- [118] P. Lennemann, Y. P. Korkolis, and A. E. Tekkaya, “Influence of changing loading directions on damage in sheet metal forming,” *Advances in Industrial and Manufacturing Engineering*, vol. 8, p. 100139, 2024.
- [119] R. Ku and T. Johnston, “Fracture strength of mgo bicrystals,” *Philosophical Magazine*, vol. 9, no. 98, pp. 231–247, 1964.
- [120] R. W. Rice, “Cao: II, properties,” *Journal of the American Ceramic Society*, vol. 52, no. 8, pp. 428–436, 1969.

Chapter 5

Publication 1

On the automated characterisation of inclusion-induced damage in 16MnCrS5 case-hardening steel

M.A. Wollenweber¹, C.F. Kusche¹, T. Al-Samman¹, S. Korte-Kerzel¹

¹Institute of Physical Metallurgy and Materials Physics, RWTH Aachen University,
Germany

Advances in Industrial and Manufacturing Engineering 2023, Volume 7, 100123

Received: 30 March 2023

Accepted: 11 June 2023

**The original article was published under the terms of the Creative Commons
Attribution-NonCommercial-No Derivatives License (CC BY NC ND).**

Maximilian A. Wollenweber: Methodology, Formal analysis, Investigation, Writing -
Original Draft

Carl F. Kusche: Supervision, Writing - Review & Editing. Talal Al-Samman:
Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition.

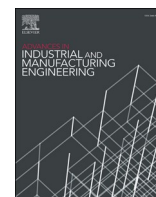
Sandra Korte-Kerzel: Conceptualisation, Writing - Review & Editing, Supervision,
Funding Acquisition.

(Online availability: 10.1016/j.aime.2023.100123)



Contents lists available at ScienceDirect

Advances in Industrial and Manufacturing Engineering

journal homepage: www.sciencedirect.com/journal/advances-in-industrial-and-manufacturing-engineering

On the automated characterisation of inclusion-induced damage in 16MnCrS5 case-hardening steel

Maximilian A. Wollenweber^{*}, Carl F. Kusche, Talal Al-Samman, Sandra Korte-Kerzel*Institute for Physical Metallurgy and Materials Physics, RWTH Aachen University, Aachen, Germany*

ARTICLE INFO

Keywords:

Damage

Machine learning

Manganese sulphide

Steel

In-situ

ABSTRACT

Manganese sulphide inclusions are commonly found in steels and known to facilitate the formation of deformation-induced damage sites in the form of voids during cold forming. These damage sites either exist as cracks, splitting the inclusion in two parts, or as delamination, separating the inclusion from the surrounding steel matrix. Both negatively influence the longevity of components, especially under cyclic loading. The analysis of damage is inherently scale-bridging, ranging from deteriorated global mechanical properties of the finished part, over the damage behaviour of individual inclusions, to the local description of individual voids. In this work, we set out to devise an analysis approach gathering information on all these scales. To this end, we conducted in-situ tensile tests while acquiring high resolution SEM panoramic images and analysed them with two neural networks, trained for this work, to detect damage sites with respect to the inclusions at which they nucleated. We find that the main damage mechanism during tensile deformation parallel to the length of inclusions is cracking and that damage evolution is equally influenced by void nucleation and void growth in the observed range of deformation. By focussing on the damaging behaviour of different inclusions, we show that the position of inclusions in the microstructure influences the resulting damage evolution and that the vicinity of pearlite bands leads to decreased damage formation.

1. Introduction

The desired geometry and mechanical properties of final products are commonly achieved during forming (Gronostajski et al., 2019; Jeswiet et al., 2008). Cold forming, which is important for obtaining a greater strength-to-weight ratio, bears the risk of initiating microstructural damage in the form of voids, due to heterogeneous properties of different microstructural constituents (Schowtjak et al., 2020; Hoefnagels et al., 2015; Tasan et al., 2014). In this respect, a large amount of research has been dedicated to understanding and preventing the nucleation of damage sites during deformation, as they compromise the material's mechanical properties (Tekkaya et al., 2017, 2020; Hering and Tekkaya, 2020; Gitschel et al., 2023). A relevant example for this is case-hardening steels, often used to manufacture gears and axles. In these materials, damage induced by non-metallic inclusions (e.g. manganese sulphide) has been the subject of intense research (Tinoco et al., 2022; Liu et al., 2019; Maciejewski, 2015). These inclusions can be beneficial, as they increase the machineability of the material and reduce hot shortness, but on the other hand they also introduce

mechanical anisotropy (Ånmark et al., 2015; Diederichs and Bleck, 2006). Additionally, during forming processes, these inclusions are harder to deform than the surrounding matrix, which leads to plastic incompatibility and the formation of micro voids (Matsuno et al., 1985; Kusche et al., 2020). Such small voids do not immediately give rise to failure but rather facilitate the formation of macroscopic cracks during service under cyclic loading, drastically reducing the longevity of products (Tekkaya et al., 2020; Moehring and Walther, 2020a, 2020b; Bruce et al., 2016). In the past, many researchers have already identified this problem and successfully focused on decreasing the number of voids in the microstructure by changing the load path during extrusion in terms of varying the deformation strains and the die geometry (Gitschel et al., 2023; Hering et al., 2020). However, because of their small size and random distribution in the microstructure, inclusions pose a challenge with respect to the associated damage analysis.

The majority of methods used to quantify microstructural damage at inclusion sites is either focussed on a high-resolution analysis, lacking sufficient statistics, or on sampling of large areas, suffering from low precision and lack of local information on individual damage sites

^{*} Corresponding author.E-mail address: wollenweber@imm.rwth-aachen.de (M.A. Wollenweber).

(Tasan et al., 2012). Combining the positive aspects of current methods regarding statistical relevance and local damage characteristics can be achieved by implementing a high-resolution large-scale analysis with high throughput based on scanning electron microscopy (SEM) and deep learning, as was demonstrated by Kusche et al. (2019) and Medghalchi et al. (2020). Hering et al. (2020) have reported a high-resolution method that acquires backscattered electron (BSE) images of large sample areas and correlates them with corresponding EDX measurements to quantify damage in the form of voids in the microstructure. This method is very precise in terms of the identification of the inclusion species, but also time-consuming and cannot distinguish damage sites by their initiation mechanism. In the current work, we propose a method that utilises secondary electron (SE) images and machine learning algorithms to detect the inclusions and damage sites in the microstructure and classifies the detected voids with respect to their size and underlying mechanism. The analysis consists of two sequential steps: initially detecting the inclusions within the matrix followed by the subsequent classification of damage sites associated with these inclusions. We set out to improve the speed of damage quantification while simultaneously acquiring more information about the damage initiation mechanism in an attempt to highlight critical features of the load path and microstructure in relation to the formation of damage. To this end we use the proposed method to analyse the damage behaviour of a 16MnCrS5 case-hardening steel subjected to quasi in-situ tensile tests with the aim to quantify global and local damage parameters along the strain path.

2. Materials and methods

2.1. Material

The investigations in this work were performed on a 16MnCrS5 case-hardening steel. The microstructure of this steel consists of elongated manganese sulphide (MnS) inclusions, in a soft ferrite matrix with stronger pearlite bands (Umemoto et al., 2003). Such MnS inclusions have been reported in monocrystalline and polycrystalline form (Liu et al., 2019; Wang et al., 2020). The heterogeneous microstructure of the investigated 16MnCrS5 steel can be observed via SE imaging in the etched state. Under these conditions the mean diameter of the ferrite grains was estimated to be approximately 10 μm and the width of the pearlite bands to be ranging from 5 to 20 μm . If the sample is unetched, pearlite, ferrite and cementite cannot be clearly distinguished from each other. However, for the detection of the MnS inclusions and related damage sites, it is sufficient to use unetched samples. Inclusions show a darker grey contrast than the steel matrix, and voids appear almost black. In Fig. 1, two inclusions with damage sites are shown in an unetched steel matrix. We distinguished between two ways in which voids can initiate due to the influence of inclusions: either they are created by forming a crack through a MnS inclusion, or by local decohesion at the interface between the inclusion and the matrix surrounding

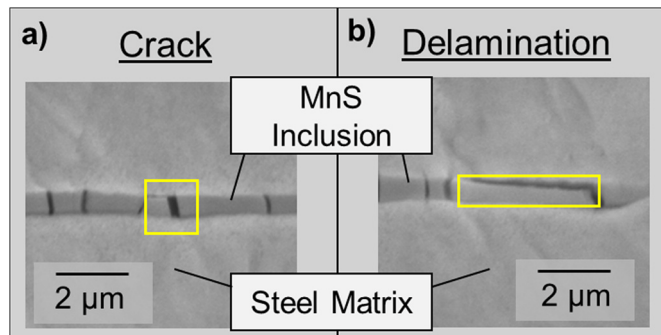


Fig. 1. SE micrograph depicting the microstructure of analysed 16MnCrS5 showing MnS inclusions with elongated shape and the two damage types distinguished in this work.

it. In this work, these mechanisms are referred to as “crack” and “delamination” respectively.

2.2. Sample preparation

To achieve a flat surface and enable damage detection in the deformed microstructure, a tensile sample was ground and polished once before the first panoramic image was taken. This was done using diamond suspensions up to 1 μm , followed by a final polishing step in a 0.25 μm oxide polishing solution (OPS) to further improve the surface quality. A second sample was metallographically prepared after it was deformed and additionally etched for 5 s in 1% Nital etching solution.

2.3. Quasi in-situ tensile tests

To study the damage behaviour induced by the MnS inclusions during deformation and to appraise the performance of the developed damage quantifying algorithm, quasi in-situ tensile tests were performed on a sample machined out of 16MnCrS5 rods as shown in Fig. 2. The obtained sample had a total length of 50 mm, a gauge length of 3.56 mm, and a thickness and gauge width of 1.5 mm. The sample was cut using electrical discharge machining (EDM) to minimise the deformation in the sample.

Prior to deformation, the original surface of interest was imaged on the undeformed tensile sample using high-resolution panoramic SE-imaging in a FEG-SEM (LEO 1530, Carl Zeiss Microscopy GmbH, Oberkochen, Germany). The individual images were obtained at an accelerating voltage of 20 kV, using an aperture with a size of 90 μm , with a lateral resolution of 32 nm/px (horizontal field width of 100 μm and 3072 pixels). Image stitching of the obtained images overlapping by 15% was performed using Image Composite Editor (Microsoft Corporation, Redmond, Washington), resulting in a seamless panoramic image with an area of 0.43 mm^2 for the undeformed sample. To ensure that inclusions do not move outside of the measured area due to the elongation in tensile direction, the length of the panoramic image was increased in tensile direction after each deformation step, increasing the imaged area to up to 0.46 mm^2 at the highest strain. The quasi in-situ tensile tests took place in a micro-tensile stage up to a plastic

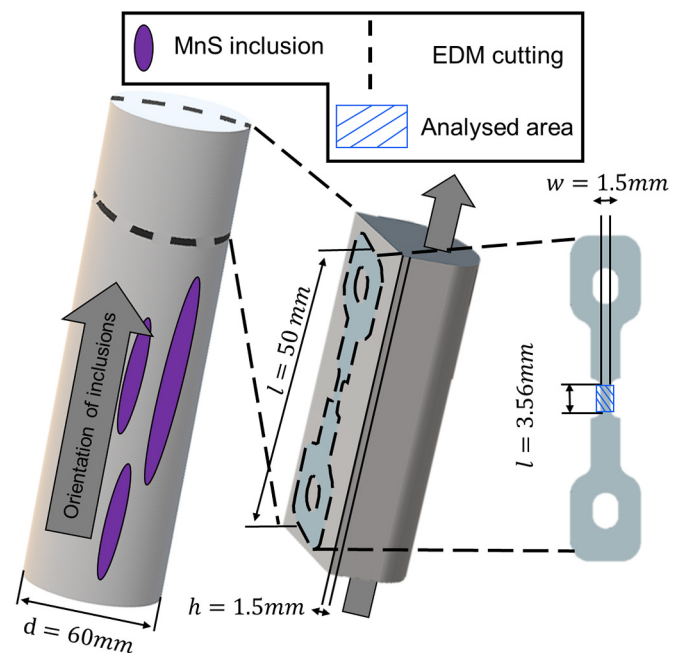


Fig. 2. Schematic illustrating the dimensions of the tensile sample and how these were cut out of 16MnCrS5 rods with respect to the orientation of the MnS inclusions.

engineering strain of 16% at a speed of 10 $\mu\text{m/s}$ (~ 0.003 1/s). After each deformation step, the deformed sample was then imaged in the same area and way to study the evolution of microstructure during deformation. The sample was deformed in a total of 5 steps, resulting in 6 panoramic images obtained at plastic engineering strains of 0%, 3%, 6%, 10%, 13% and 16%. The second sample was deformed to a strain of 16%.

2.4. Damage quantification

Detection and subsequent quantification of damage sites was conducted on unetched samples using a sequence of 2 convolutional neural networks, which were specifically trained for their tasks in this work, followed by k-means clustering. It is a commonly used strategy when developing a network used for computer vision to take an already trained network and retrain it with images necessary to accomplish its task (Doulamis et al., 2000; Yu and Seltzer, 2011; Pham, 2020). This was also done for the Faster R-CNN convolutional networks with the ResNet50 architecture used in this work, that were already pretrained on the COCO 2017 dataset and acquired from the TensorFlow model zoo (Huang et al., 2017). The first neural network was trained on images of the microstructure at various strain levels acquired from tensile test specimens to separate the MnS inclusions that damage from the plastically deforming matrix. The resulting areas of interest found by this network feature complete inclusions, independent of the fact whether these inclusions show damage in the form of voids or not. Fig. 3 shows an example of this procedure, where areas with inclusions in the 16% deformed microstructure are outlined in green and two inclusions are magnified. In a similar manner, the second neural network was trained on images of deformed inclusions to classify corresponding damage sites. The networks were trained on 6 samples with varying degree of damage containing a total of 358 inclusions and 415 damage sites that were manually labelled. If damage sites are present in the output of the first network, they are isolated, and the damage initiation mechanism is classified as either cracking or delamination. An example of detected and classified damage sites is also shown in Fig. 3 (highlighted in yellow). These images were further processed by using two greyscale thresholds to distinguish between the actual damage site and the remaining parts of the inclusion and the steel matrix around the inclusion. To automate this thresholding and enable measurement of the individual voids, a K-means clustering algorithm (Hartigan and Wong, 1979) was employed, as highlighted in purple in Fig. 3. The threshold analysis of greyscale images distinguishes voids (shown in black) from inclusions and the surrounding matrix, which are depicted in grey and a lighter grey respectively. This colour coding can be observed in the clustered image (left image highlighted in purple in Fig. 3), allowing for the extraction of pixels belonging to the void (right image highlighted in purple in Fig. 3). Using this procedure makes it also possible to gather

local information, such as the area, the position, the initiation mechanism per void and also the number of voids per inclusions. Combining the individual information of all found voids also provides information about global damage parameters such as Void Area Fraction (VAF), Number of Voids (NoV) and the dominant initiation mechanism.

3. Results

Analysis of damage comprised 6 analysed panoramic images at the plastic engineering strains of 0%, 3%, 6%, 10%, 13% and 16%. The results are shown in Table 1 in terms of the (i) Void Area Fraction (VAF) given in parts per million (ppm), which relates the area covered by voids to the total imaged area, (ii) the Number of Voids (NoV) per unit area and (iii) the ratio of the number of detected cracks to the number of detected delamination sites. Additionally, the evolution of these damage parameters is shown in Fig. 4 as a function of strain. As evident from Fig. 4, both VAF and NoV reveal an increasing trend with strain.

In addition to the global parameters, local damage characteristics describing the evolution of individual damage sites were obtained from the analysis. This is shown in Fig. 5 as an example for one individual MnS inclusion in the undeformed state and at 6% and 16% strain. Similar to the global damage parameters, the local number and area of voids increase continuously with deformation.

Even though damage nucleation is a local microstructural event, the total number of sites for each damage mechanism can be scrutinised in order to find out which mechanism is globally dominant during the course of deformation. This is shown in Table 1, which reveals that the ratio of the number of cracks relative to delamination sites rises with strain.

4. Discussion

4.1. Global damage behaviour

Using the described method for damage analysis during tension in the direction of the largest MnS dimension allows for damage quantification on both the global scale, focussing on the entire panoramic image, as well as on the local scale, focussing on individual inclusions and

Table 1

Void area fraction, number of damage sites/voids (NoV) and ratio of cracks to delamination sites as a function of plastic strain.

Plastic strain (%)	0	3	6	10	13	16
Void area fraction (ppm)	0.6	12.6	22.7	38.1	63.7	85.2
NoV ($10^2/\text{mm}^2$)	0.10	1.45	2.56	4.89	6.37	7.77
Cracks/Delamination	0.3	3.8	9.5	8.7	15.7	14.2

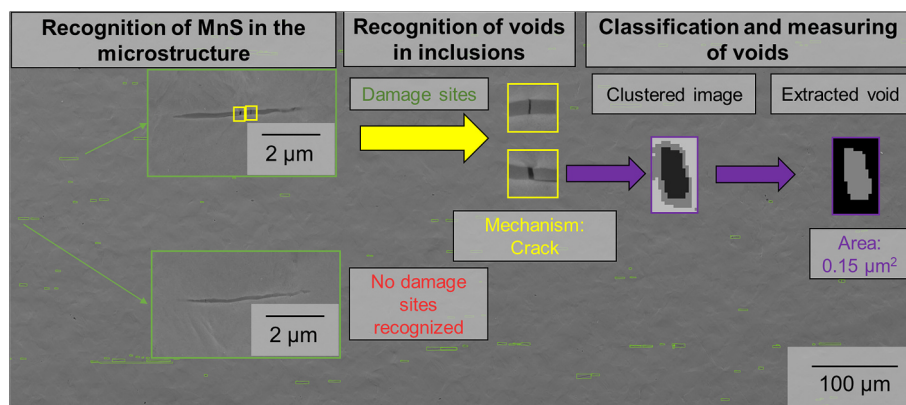


Fig. 3. Workflow of inclusion detection (green), damage detection and classification (yellow) and quantification (purple) using two neural networks and a K-means clustering algorithm. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

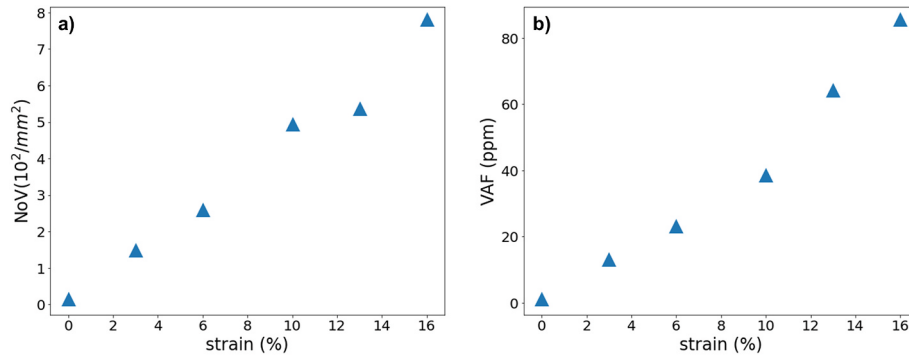


Fig. 4. Change of NoV (a), VAF (b) over the course of deformation during quasi in-situ tensile testing.

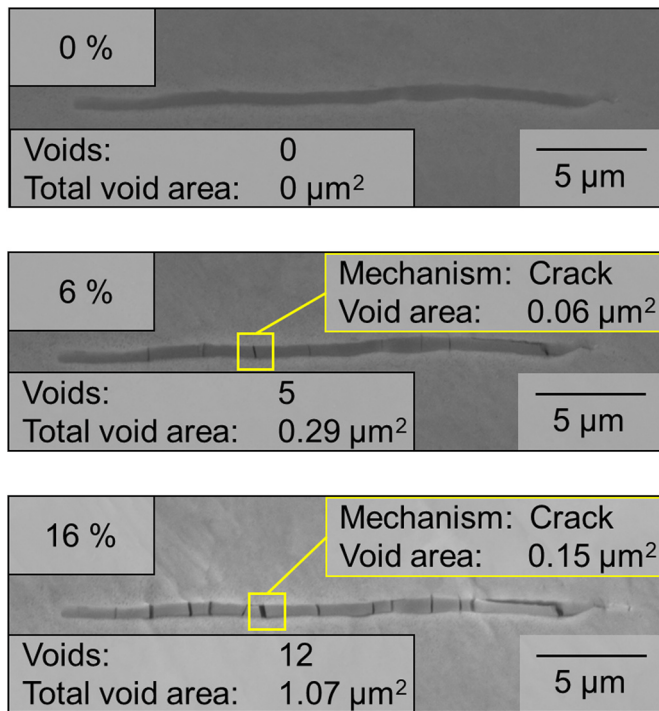


Fig. 5. Quasi in-situ observations of the damage formation and evolution at a MnS inclusion at 0%, 6% and 16% tensile strains. An example of the quantified damage parameters is shown for one individual void (highlighted in yellow) and for the whole inclusion. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

voids. On the global scale, a clear trend of increasing damage parameters is observed during successive straining, as both NoV and VAF increase similarly (Fig. 4). If one of these damage parameters was to increase faster in relation to the other, this would be indicative of either a nucleation or growth-controlled process. For the range of strains investigated here, we cannot identify a dominance of one parameter or a transition to a clearly accelerated increase in area or number of voids. This is consistent with the results of the determined mean void area, analysed at every deformation step. For a growth-dominated increase of VAF (i.e. facilitated growth of a small number of voids), the mean void size should increase, whereas for a nucleation-dominated process (high nucleation rate and restricted growth), it would become smaller. The mean void size determined in this work shows no specific trend as a function of strain as shown Fig. 6.

This confirms the previous assessment based on VAF and NoV, namely that damage formation is neither dominated by nucleation nor by growth, i.e. they both affect damage in equal parts. This observation

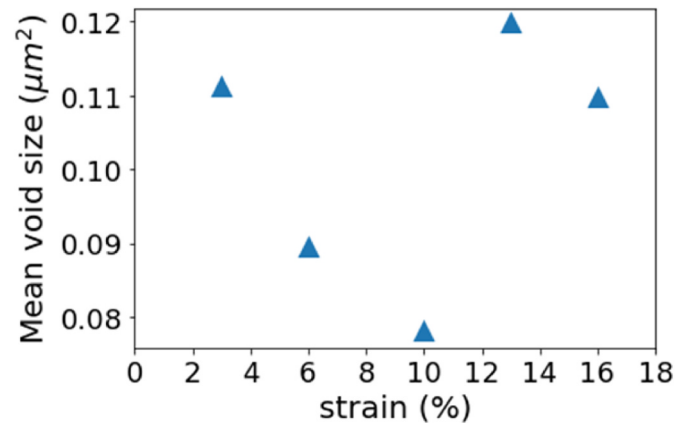


Fig. 6. Change of the mean void size during quasi in-situ tensile straining.

agrees with related studies in the literature, even if the underlying microstructures are often very different, in particular either lacking mechanical contrast between different phases or a more continuous network of hard and soft phases. Bao and Wierzbicki concluded that failure of metals in the range of low positive triaxialities is caused by a mixed mechanism consisting of void growth and shear bands (Bao and Wierzbicki, 2004). Kusche et al. reported that the triaxiality mainly influences damage growth, while the applied strain mainly influences the damage nucleation in DP800 steel (Kusche et al., 2021). Applying this knowledge to the present results means that the global parameter of stress triaxiality was facilitating growth in such a way that it had a similar contribution to the VAF increase caused by nucleation. It further suggests that a transition in the damage behaviour can take place, i.e. the damage evolution would be dominated by growth if the global triaxiality is to increase, e.g. by necking of the tensile sample at larger strains.

4.2. Local damage nucleation and evolution

4.2.1. Prevalence of damage initiation mechanisms

Because the used method of damage quantification is of sufficient local resolution, the damage characteristics at individual inclusion sites and even of individual damage sites can be compared easily for one given strain or for different strains. The latter is considered in Table 1, where the continuously increasing ratio of inclusion cracks to delamination damage at the inclusion/matrix interface shows that inclusion internal cracking is the dominant damage initiation mechanism during uniaxial tension along the direction of the largest MnS dimension. The reason for this can be found in the stresses acting on the inclusion since the driving force for damage formation is produced by the applied tensile stress irrespective of the initiation mechanism. However, the

applied tensile stress subjects the interfaces of inclusions to shear stress in this direction of straining, i.e. parallel to the longitudinal axis of the inclusions, leading to limited formation of delamination damage, because these shear stresses are not promoting void growth and the transverse contraction of the sample even leads to forces counteracting the growth of these voids. As a result, delamination damage has a low prevalence and the delamination damage sites that are present have either nucleated at cracks (cf. Figs. 5 and 16% strain) or at the edges of inclusions where the adhesion of the interface was not sufficient and “fibre” pull-out is taking place as shown in Fig. 7.

This suggests that the dominant mechanism can be influenced by changing the loading direction. When the loading direction is adjusted in a way that the sample is strained in tension perpendicular to the direction of the largest MnS dimension, it is expected that delamination will be the dominant damage initiation mechanism, because normal forces are acting on the interface, promoting void nucleation and growth by delamination.

Micro-cracks, on the other hand, are easily initiated within the deformed inclusions at $\sim 90^\circ$ from the direction of their largest dimension if the stress is applied parallel to this direction. Here the driving force, the applied tensile stress, produces normal stresses at the crack interfaces, and the sufficient adhesion of the inclusion additionally leads to the limited prevalence of “fibre” pull-out. A closer investigation of these damage sites reveals that internal cracks are never in immediate vicinity to each other as shown in Fig. 8. The distance between neighbouring damage sites was measured for 12 inclusions in the last deformation step at 16% strain, resulting in a total of 47 spacings in the direction of the largest MnS dimension, measured from the centre point of one crack to the next. The estimated values spanned over a wide range reaching up to $5.8 \mu\text{m}$, but never reaching a distance lower than $0.6 \mu\text{m}$. The distribution of crack distances also spanned over a wide range inside of individual inclusions, as also shown in Fig. 8, for a particularly long MnS inclusion with a total length of $37 \mu\text{m}$.

There are two obvious reasons for a minimum spacing between cracks related to the origin of crack nucleation: brittle cracks do not only require a sufficient crack driving force, such that crack growth promotes relaxation of elastically stored energy, but also a nucleation site containing a critical flaw. Crack nucleation sites may result from plastic deformation within the MnS inclusion itself or from stress concentrations formed at the interface due to plasticity in the surrounding matrix, as known from fibre composite materials (Jollivet et al., 2013). Slip and cleavage on specific planes in the MnS phase are strongly orientation dependent (Kusche et al., 2020) and if these planes were the sole origin of the observed cracks, cracks would be expected to strongly scatter in orientation relative to the tensile axis. This is, however, not observed in the current study, as cracks are always nucleated at $\sim 90^\circ$ to the longitudinal axis of the inclusion. If slip in the MnS phase was to occur at a considerable rate, shearing of the particles would be expected, but is also not observed. Crack initiation from stress concentration sites owing to matrix deformation should, in quasi in-situ straining with a free surface,

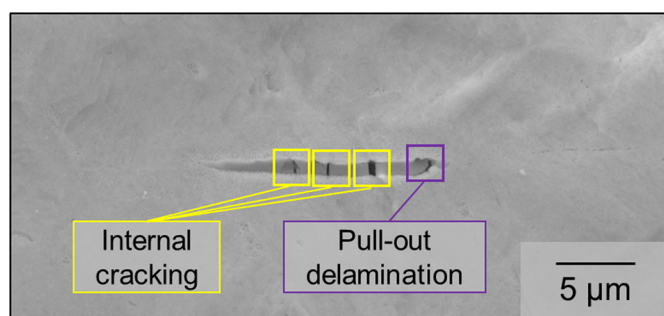


Fig. 7. Inclusion showing internal cracks and pull-out delamination at 16% strain.

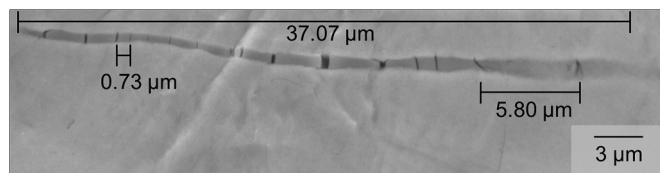


Fig. 8. Example of a MnS inclusion with multiple damage sites (internal cracks) in an unetched sample at 16% strain. The maximum spacing between two cracks along the direction of the largest MnS dimension is $5.8 \mu\text{m}$ and the minimum spacing $0.73 \mu\text{m}$.

correlate with the formation of slip traces in the vicinity of cracks, at least where a slip component normal to the surface is present. This is in fact observed here (Fig. 8), suggesting that stress concentrations in the matrix act as damage nucleation sites. However, more detailed in-situ work using additional methods such as electron backscatter diffraction (EBSD) to image dislocation induced lattice rotations before crack initiation will be required to clearly show that localisation of plasticity in the ferrite matrix at the inclusion interface is in fact the initial step. As the aim of this study was to provide a high throughput analysis, EBSD could not be included. However, the presented method, including identification of the MnS inclusions by a neural network, could be linked to a further automated work flow allowing site specific EBSD mapping during straining in future work. The combination of stress concentrations from the matrix acting as damage nucleation sites and normal stress acting as a driving force, leads to the observed distribution of crack spacings. If normal stress alone was the only driver of cracking, more regular crack spacings are expected, as in fibre composites (Cuyper and Wastiels, 2006), but are not observed in this work.

4.2.2. Damage evolution at individual inclusions

Considering the mean damage size during ongoing deformation irrespective of the dominant initiation mechanism (cf. Fig. 5), it is evident that the size of individual damage sites increases, reproducing the discussed global damage behaviour at a local scale. Additionally, it is seen that new voids nucleate even at later stages of deformation, as shown in Fig. 5. These new voids decrease the mean void size in the sample compared to a case, where the same void area is attained via fewer voids. Interestingly, the mean void size increases for this specific inclusion in Fig. 5, as it rises from $0.06 \mu\text{m}^2$ at a strain of 6% to $0.09 \mu\text{m}^2$ at a strain of 16%. However, the global mean void size does not increase, leading to the conclusion that other inclusions must show stronger nucleation than growth. Another comparison of the damage behaviour is shown in Fig. 9 for different inclusions at the same strain of 16%. The featured inclusions show different levels of damage comprising (a) no damage at all, (b) nucleated micro-voids and (c) grown micro-voids. This different progress of damage at different inclusions is likely the reason for the ambiguous trend of the void size at the measured strains between zero and 16%.

As evident by the wavy microstructure in the matrix around the damaged inclusions (Fig. 9 b and c), it is likely that the local deformation behaviour of the softer matrix affects the evolution of damage in surrounded inclusions. In Fig. 9 c, the matrix appears strongly deformed around the inclusion with grown voids, while the matrix in Fig. 9 a shows almost no signs of deformation and the inclusion itself shows no voids. The deformation behaviour of the matrix is determined by its crystallographic orientation, and in the current case, also by its local phase distribution, which remain concealed in Fig. 9. To shed light on this aspect, a deformed sample was etched to reveal the ferritic-pearlitic matrix. Since the pearlite structure has different mechanical properties than ferrite, they are expected to deform differently, which should have implications for the damage formation at inclusions nearby, suggesting that the position of an inclusion in the matrix plays a major role, as postulated by Qayyum et al. (2022). Fig. 10 depicts an example of a MnS inclusion that is partially encompassed by pearlite and ferrite. The part

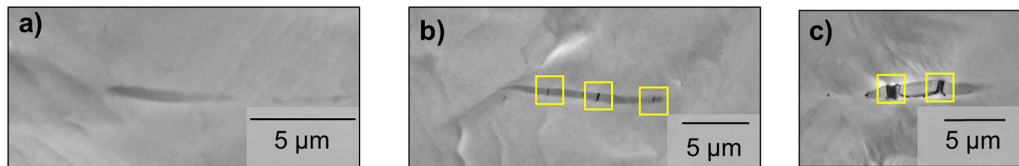


Fig. 9. Differences of damage extent between three inclusions at a global strain of 16%. Inclusions can show no damage at all (a)), only nucleation (b)) and grown voids (c)).

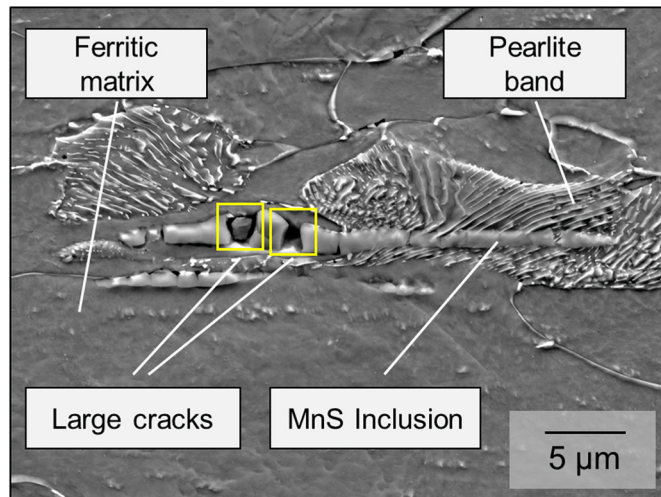


Fig. 10. Etched 16MnCrS5 microstructure showing a MnS inclusion that is partially engulfed by pearlite (lamellar structure). The outlined areas show greater crack opening in the parts of the inclusion that are surrounded by the ferrite matrix, i.e. not the pearlite structure.

of the inclusion next to pearlite experienced less damage compared to the parts in the ferrite matrix, which exhibit a greater crack opening. A statistical assessment of this observation is needed but at this point not possible, because the introduced automated method cannot reliably handle an etched microstructure yet. However, from Fig. 10 it is reasonable to assume that damage of MnS inclusions in this material can be reduced by controlling the pearlite content in the microstructure.

As discussed for the prevalence of different mechanisms, it was suggested that stress concentrations in the matrix around the inclusion act as damage nucleation sites. As pearlite shows less stress concentration at the inclusion interface than the ferrite matrix, because it is harder to deform, it is suggested that the discussed average spacing between damage sites, and hence their number density also depends on whether the microstructural surrounding is ferrite or pearlite. For the shown example in Fig. 8 it is evident that a small spacing between two neighbouring damage sites exists in the region, where the matrix (most likely ferrite) exhibited a rough wavy appearance and deformation steps. On the other hand, less deformation is observed on the right-hand side of the SE image, where a maximum spacing between damage sites was measured and where pearlite it is expected.

5. Conclusions

In the current work, 16MnCrS5 steel, containing elongated MnS inclusions, was subjected to quasi in-situ tensile tests along the direction of the largest inclusion dimension to investigate microstructural damage on global and local (mesoscopic) scales. We developed an automated damage quantification method that utilises panoramic, high-resolution SE imaging and machine learning algorithms trained to detect and classify damage types at MnS inclusions. The following conclusions can be drawn from the obtained results.

- The applied method of automated damage analysis using two neural networks allowed the determination of global damage properties, such as the number of voids and the void area fraction, and provided valuable local information regarding the individual void size and damage initiation mechanism.
- The method also allowed for useful comparisons of individual damage sites occurring at inclusions in the context of their initiation and evolution as a function of strain or as a function of their microstructural surrounding.
- With increasing strain there was an increase in damage reflected by an upsurge of damage parameters (NoV and VAF). This was influenced by continuous events of void nucleation and growth during deformation up to a maximum strain of 16%. At larger strains (not investigated in the current work), the mode of damage evolution is expected to be predominantly controlled by growth due to a local increase of stress triaxiality during sample necking.
- The extent of damage at individual MnS inclusions was observed to depend on the deformation behaviour of the surrounding ferrite/pearlite matrix. Inclusion parts surrounded by the ferrite matrix showed greater crack opening than other parts near pearlite. An observed uneven distribution of internal cracks that were either separated by $< 1 \mu\text{m}$ in case of a strongly deformed matrix or several micrometres in case of a less deformed matrix, is suggested to be dependent on the surrounding pearlite as well.
- Under uniaxial tensile loading along the direction of the largest MnS dimension, internal cracking of MnS inclusions is the major damage initiation mechanism compared to delamination damage, i.e. separation of the inclusion from the surrounding steel matrix. This can change if tensile loading was applied perpendicular to the direction of the largest MnS dimension.
- The presented information suggests that additional parameters have to be considered when designing damage tolerant processes and materials, such as the loading direction with respect to the largest dimension of elongated inclusions and the local microstructural environment surrounding inclusions.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgments

The authors express their gratitude to the Deutsche Forschungsgemeinschaft (DFG) for financial support in context of the Collaborative Research Centre CRC/Transregio 188/2 “Damage Controlled Forming Processes”, project B02, project no. 278868966.

References

- Ånmark, N., Karasev, A., Jönsson, P.G., 2015. The effect of different non-metallic inclusions on the machinability of steels. *Materials* 8 (2), 751–783.
- Bao, Y., Wierzbicki, T., 2004. On fracture locus in the equivalent strain and stress triaxiality space. *Int. J. Mech. Sci.* 46 (1), 81–98.
- Bruce, T., Long, H., Slatter, T., Dwyer-Joyce, R., 2016. Formation of white etching cracks at manganese sulfide (MnS) inclusions in bearing steel due to hammering impact loading. *Wind Energy* 19 (10), 1903–1915.
- Cuyper, H., Wastiels, J., 2006. Stochastic matrix-cracking model for textile reinforced cementitious composites under tensile loading. *Mater. Struct.* 39, 777–786.
- Diederichs, R., Bleck, W., 2006. Modelling of manganese sulphide formation during solidification, part I: description of MnS formation parameters. *Steel Res. Int.* 77 (3), 202–209.
- Doulamis, A.D., Doulamis, N.D., Kollias, S.D., 2000. On-line trainable neural networks: improving the performance of neural networks in image analysis problems. *IEEE Transact. Neural Networks Learn. Syst.* 11 (1), 137–155.
- Gitschel, R., Hering, O., Schulze, A., Erman Tekkaya, A., 2023. Controlling damage evolution in geometrically identical cold forged parts by counterpressure. *J. Manuf. Sci. Eng.* 145 (1), 011011.
- Gronostajski, Z., Pater, Z., Madej, L., Gontarz, A., Lisecki, L., Łukaszek-Sotek, A., Łukasz, J., Mróz, S., Muskalski, Z., Muzykiewicz, W., 2019. Recent development trends in metal forming. *Arch. Civ. Mech. Eng.* 19 (3), 898–941.
- Hartigan, J.A., Wong, M.A., 1979. Algorithm AS 136: a k-means clustering algorithm. *J. Roy. Stat. Soc.: series c* 28 (1), 100–108.
- Hering, O., Tekkaya, A.E., 2020. Damage-induced performance variations of cold forged parts. *J. Mater. Process. Technol.* 279, 116556.
- Hering, O., Dunlap, A., Tekkaya, A.E., Aretz, A., Schwedt, A., 2020. Characterization of damage in forward rod extruded parts. *Int. J. Material Form.* 13, 1003–1014.
- Hoefnagels, J.P., Tasan, C.C., Maresca, F., Peters, F.J., Kouznetsova, V.G., 2015. Retardation of plastic instability via damage-enabled microstrain delocalization. *J. Mater. Sci.* 50 (21), 6882–6897.
- Huang, J., Rathod, V., Sun, C., Zhu, M., Korattikara, A., Fathi, A., Fischer, I., Wojna, Z., Song, Y., Guadarrama, S., 2017. Speed/accuracy trade-offs for modern convolutional object detectors. In: *Proceedings of the IEEE Conference on Computer Vision and Pattern Recognition*, pp. 7310–7311.
- Jeswiet, J., Geiger, M., Engel, U., Kleiner, M., Schikorra, M., Duflo, J., Neugebauer, R., Bariani, P., Bruschi, S., 2008. Metal forming progress since 2000. *CIRP Journal of Manufacturing Science and Technology* 1 (1), 2–17.
- Jollivet, T., Peyrac, C., Lefebvre, F., 2013. Damage of composite materials. *Procedia Eng.* 66, 746–758.
- Kusche, C., Reclik, T., Freund, M., Al-Samman, T., Kerzel, U., Korte-Kerzel, S., 2019. Large-area, high-resolution characterisation and classification of damage mechanisms in dual-phase steel using deep learning. *PLoS One* 14 (5), e0216493.
- Kusche, C.F., Gibson, J.S.-L., Wollenweber, M.A., Korte-Kerzel, S., 2020. On the mechanical properties and deformation mechanisms of manganese sulphide inclusions. *Mater. Des.* 193, 108801.
- Kusche, C.F., Pütz, F., Münstermann, S., Al-Samman, T., Korte-Kerzel, S., 2021. On the effect of strain and triaxiality on void evolution in a heterogeneous microstructure—A statistical and single void study of damage in DP800 steel. *Mater. Sci. Eng.* 799, 140332.
- Liu, X.-g., Wang, C., Gui, J.-t., Xiao, Q.-q., Guo, B.-f., 2019. Effect of MnS inclusions on deformation behavior of matrix based on in-situ experiment. *Mater. Sci. Eng.* 746, 239–247.
- Maciejewski, J., 2015. The effects of sulfide inclusions on mechanical properties and failures of steel components. *J. Fail. Anal. Prev.* 15, 169–178.
- Matsuno, F., Nishikida, S.-i., Ikesaki, H., 1985. Mechanical properties of manganese sulphides in the temperature range between room temperature and 1000° C. *Transaction of the Iron and Steel Institute of Japan* 25 (9), 989–998.
- Medghalchi, S., Kusche, C.F., Karimi, E., Kerzel, U., Korte-Kerzel, S., 2020. Damage analysis in dual-phase steel using deep learning: transfer from uniaxial to biaxial straining conditions by image data augmentation. *Jom* 72 (12), 4420–4430.
- Moehring, K., Walther, F., 2020a. Performance-related characterization of forming-induced initial damage in 16MnCrS5 steel under a torsional forward-reverse loading path at LCF regime. *Materials* 13 (11), 2463.
- Moehring, K., Walther, F., 2020b. Load direction-dependent influence of forming-induced initial damage on the fatigue performance of 16MnCrS5 steel. *Materials* 13 (12), 2680.
- Pham, T.D., 2020. A comprehensive study on classification of COVID-19 on computed tomography with pretrained convolutional neural networks. *Sci. Rep.* 10 (1), 1–8.
- Qayyum, F., Umar, M., Elagin, V., Kirschner, M., Hoffmann, F., Guk, S., Prahl, U., 2022. Influence of non-metallic inclusions on local deformation and damage behavior of modified 16MnCrS5 steel. *Crystals* 12 (2), 281.
- Schowitz, A., Wang, S., Hering, O., Clausmeyer, T., Lohmar, J., Schulte, R., Ostwald, R., Hirt, G., Tekkaya, A.E., 2020. Prediction and analysis of damage evolution during caliber rolling and subsequent cold forward extrusion. *J. Inst. Eng. Prod.* 14, 33–41.
- Tasan, C., Hoefnagels, J., Geers, M., 2012. Identification of the continuum damage parameter: an experimental challenge in modeling damage evolution. *Acta Mater.* 60 (8), 3581–3589.
- Tasan, C.C., Hoefnagels, J.P., Diehl, M., Yan, D., Roters, F., Raabe, D., 2014. Strain localization and damage in dual phase steels investigated by coupled in-situ deformation experiments and crystal plasticity simulations. *Int. J. Plast.* 63, 198–210.
- Tekkaya, A.E., Khalifa, N.B., Hering, O., Meya, R., Myslicki, S., Walther, F., 2017. Forming-induced damage and its effects on product properties. *CIRP Annals* 66 (1), 281–284.
- Tekkaya, A., Bouchard, P.-O., Bruschi, S., Tasan, C., 2020. Damage in metal forming. *CIRP Annals* 69 (2), 600–623.
- Tinoco, H.A., Fintova, S., Heikkilä, I., Herrero, D., Vuoristo, T., Dlouhy, I., Hutar, P., 2022. Experimental and numerical study of micromechanical damage induced by MnS-based inclusions. *Mater. Sci. Eng.* 856, 144009.
- Umemoto, M., Todaka, Y., Tsuchiya, K., 2003. Mechanical Properties of Cementite and Fabrication of Artificial Pearlite. *Materials Science Forum*, pp. 859–864.
- Wang, C., Liu, X.-g., Gui, J.-t., Du, Z.-l., Xu, Z.-f., Guo, B.-f., 2020. Effect of MnS inclusions on plastic deformation and fracture behavior of the steel matrix at high temperature. *Vacuum* 174, 109209.
- Yu, D., Seltzer, M.L., 2011. Improved Bottleneck Features Using Pretrained Deep Neural Networks, Twelfth Annual Conference of the International Speech Communication Association.

Chapter 6

Publication 2

On the damage behaviour in dual-phase DP800 steel deformed in single and combined strain paths

M.A. Wollenweber¹, S. Medghalchi¹, L.R. Guimaraes¹, N. Lohrey¹, C.F. Kusche¹, U. Kerzel², T. Al-Samman¹, S. Korte-Kerzel¹

¹Institute of Physical Metallurgy and Materials Physics, RWTH Aachen University, Germany

²Faculty of Georesources and Materials Engineering, RWTH Aachen University, Germany

Materials & Design 231 (2023) 112016

Received: 14 March 2023

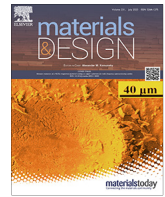
Accepted: 12 May 2023

The original article was published under the terms of the Creative Commons Attribution-NonCommercial-No Derivatives License (CC BY NC ND).

Maximilian A. Wollenweber: Methodology, Formal analysis, Investigation, Writing - Original Draft

Setareh Medghalchi: Software, Writing - Review & Editing. Luiz R. Guimaraes: Investigation, Writing - Review & Editing. Nicole Lohrey: Investigation, Writing - Review & Editing. Carl F. Kusche: Investigation, Writing - Review & Editing. Ulrich Kerzel: Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition. Talal Al-Samman: Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition. Sandra Korte-Kerzel: Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition.

(Online availability: [j.matdes.2023.112016](https://doi.org/10.1016/j.matdes.2023.112016))



On the damage behaviour in dual-phase DP800 steel deformed in single and combined strain paths

Maximilian A. Wollenweber^{*,a}, Setareh Medghalchi^a, Luiz R. Guimarães^a, Nicole Lohrey^a, Carl F. Kusche^a, Ulrich Kerzel^b, Talal Al-Samman^a, Sandra Korte-Kerzel^a

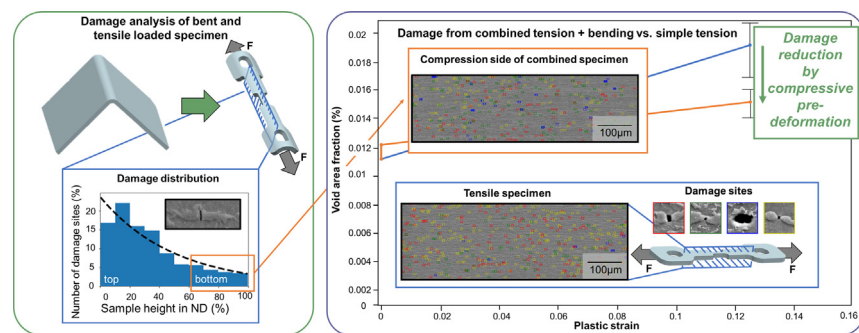
^a Institute for Physical Metallurgy and Materials Physics, RWTH Aachen University, Aachen, Germany

^b Faculty of Georesources and Materials Engineering, RWTH Aachen University, Aachen, Germany

HIGHLIGHTS

- We show that when deformation first proceeds under a negative stress triaxiality and then, due to a strain path change, under positive triaxiality, damage formation is reduced owing to a favourable effect of strain hardening on inhibiting void nucleation and growth.
- We also show that when deformation proceeds under positive triaxiality during the first and the second strain path, damage formation is promoted, despite strain hardening, because the number of voids is already high after the first strain path.
- In strain paths where deformation under positive triaxiality is followed by deformation under negative triaxiality, the damage parameters show a decrease that is likely related to a reduction of ductile damage by void closure due to negative hydrostatic pressure.
- Changing the loading direction between the first and second strain path decreases the rate of damage formation, as compared to a fixed loading direction during both strain paths.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 14 March 2023

Revised 28 April 2023

Accepted 17 May 2023

Available online 20 May 2023

Keywords:

Dual-phase steel

ABSTRACT

Ductile damage in dual-phase steels deteriorates the mechanical properties, resulting in a lowered crash-worthiness and a shorter life-time under cyclic loading. However, most works focus on the observation of damage under simple strain paths, even though work pieces are often subjected to combined strain paths during forming. In this work, we set out to characterise the damage behaviour for a DP800 steel subjected to combinations of tensile and bending loads. To this end, we use an automated approach based on machine learning to quantify damage in high-resolution SEM panoramic images, coupled with finite element modelling to determine the stress state, and high-throughput nanoindentation to determine the

* Corresponding author.

Damage
Machine Learning
Nanoindentation

strain hardening behaviour. We find a strong connection between loading direction and damage formation in both steps of the forming process. By focussing on different possible combinations of positive and negative triaxiality, we show that the combination of two deformation modes with positive triaxiality leads to a promotion of damage formation. A negative triaxiality applied after a positive triaxiality leads to void closure, reducing the number of damage sites and total void area. Importantly, deformation with negative triaxiality carried out before deformation with positive triaxiality inhibits damage nucleation and growth due to strain hardening.

© 2023 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

1. Introduction

With rising demand for better mechanical properties and increasing safety requirements, it becomes inevitable that safety-relevant structural components need to be engineered in a way that guarantees the targeted mechanical properties along the line of production up to the finished product. This can be very challenging because during forming processes, initiation and evolution of microstructural damage sites have a strong negative influence on the mechanical properties [1,2]. Due to void formation, growth and coalescence, premature ductile failure is very likely to occur, especially during cyclic loading [3,4]. This not only has a negative impact on the material's formability to achieve the desired geometry but also on its crashworthiness, of relevance for example in the automotive industry and the longevity of the part during use. As was reported in [5], the occurrence of ductile failure in high-strength multiphase steels can be related to a combination of ductile damage mechanisms and shear localization. This is particularly observed in DP steels because of their especially heterogeneous microstructure consisting of a soft ferritic matrix with hard martensitic islands inside [6,7]. The structural, chemical and mechanical contrast of these constituent phases leads to a heterogeneous stress distribution during forming, and thereby strongly impacts the material's mechanical behaviour [8,9]. Damage in DP steel has been subject to intense research in the past years, where significant knowledge regarding the damage mechanisms occurring during deformation along simple strain paths has been established [10–18]. The results of these works show that martensite cracking and interface decohesion are the main mechanisms for microstructural damage [13,19]. In that respect, some works reported that the contribution of interface decohesion to damage nucleation increases at higher strain levels [16,20,21]. One challenge in DP steel research lies in its inherent, local heterogeneity, rendering direct comparisons between different steel grades difficult. Recently, work based on the use of artificial intelligence for image analysis, has begun using damage analyses across large areas to address this issue by improving phase differentiation [22], confirming the change in dominant damage mechanism with strain and stress state [21,23], describing the reorientation of martensite cracks in biaxial straining [24] and uncovering the additional effect of triaxiality [25] and crystal orientation [26] on damage nucleation and growth.

In an industrial context, various sheet metal forming processes subject the material to a complex deformation history by a combination of (locally) different strain paths. To develop damage models that can be applied to more realistic cases, it is therefore important to mimic these complex strain paths and investigate nucleation, growth and interaction of damage sites during each step of the forming process both at a local and global scale. In literature some works concerning complex strain paths have been conducted on DP steel but with a focus on the global mechanical properties [27–29]. For this reason, we present here a systematic microstructural study of micro-

scopic damage evolution as a function of imposed strain path. The loading scenarios investigated include simple tension, simple bending and a combination of tension and bending in two different sequences. Our aim is to clarify how a changing stress-state along the strain path affects damage behaviour in each model case, and to discuss how this knowledge can be utilized to reduce damage and to support optimization of forming processes. To this end, we used machine learning to analyse panoramic images acquired in a scanning electron microscope to detect and classify damage sites in deformed samples. In addition, we employ finite-element (FE) simulations to link the damage behaviour with the strain distribution and evolution of the stress state during deformation. Lastly, we make use of high-throughput nanoindentation mappings to quantify strain hardening and link it to the damaging process. Recently this method has become more popular in literature [30–32] because of its ability to generate large statistical data sets within short time periods, making it possible to distinguish between microstructural constituents of small size because of the low indentation depth and high spatial resolution.

2. Materials and methods

2.1. Material

The investigations in this work were performed on a commercial dual-phase steel of DP800 grade. The chemical composition of this steel determined by wet-chemical analysis is given in Table 1 and the mechanical properties determined by tensile tests are shown in Table 2. Both the chemical composition and mechanical properties are taken from [33].

A typical microstructure of the investigated DP800 sheet, reproduced from the RD-ND plane of the sheet (RD, TD, ND refer to the rolling, transverse and normal directions), is shown in Fig. 1. The insets in the figure show magnified views featuring banding of the martensite phase along the RD (outlined in orange) and the ferrite matrix (outlined in blue). A typical microstructural damage site in form of a brittle crack in a martensitic band is also shown (outlined in green).

2.2. Bending tests

In order to investigate a combined strain path consisting of bending and subsequent tensile loading under quasi-static conditions, the investigated DP800 sheet material with a thickness of 1.5 mm was first cut into 60 mm × 60 mm samples using electrical discharge machining (EDM) for simple three-point bending tests. For the bending tests, a stamp with a radius of 1 mm and a die with a width of 8 mm and radii of 1 mm were used to deform the samples to a plastic equivalent strain (PEEQ) of 0.3 in bending direction, measured at the outer edge of the bending side, resulting in a bending angle of 64° (cf. Fig. 2a). Stress triaxiality during bending was estimated by FE simulations and

Table 1
Chemical composition in weight percent (%) determined by wet-chemical analysis of analysed DP steel as taken from [33] (DP2).

C	Si	Mn	P	S	Al
0.131	0.194	1.69	0.0088	0.0016	0.038
Cr	Mo	Nb	Ti	V	B
0.718	0.0035	< 0.001	0.0303	0.0027	< 0.001

Table 2
Mechanical properties of analysed DP steel measured in tensile tests as taken from [33] (DP2).

Yield stress (MPa)	UTS (MPa)	UE (%)	TE (%)
494	830	11.5	18.6

showed a maximum value of 0.57 at the outer edge of the bending side (presented later in Fig. 9). The bent samples were first used for individual analysis of damage during bending and then processed further for damage analysis after two-step bending and tensile testing. In total, the damage behaviour of three bending samples was analysed.

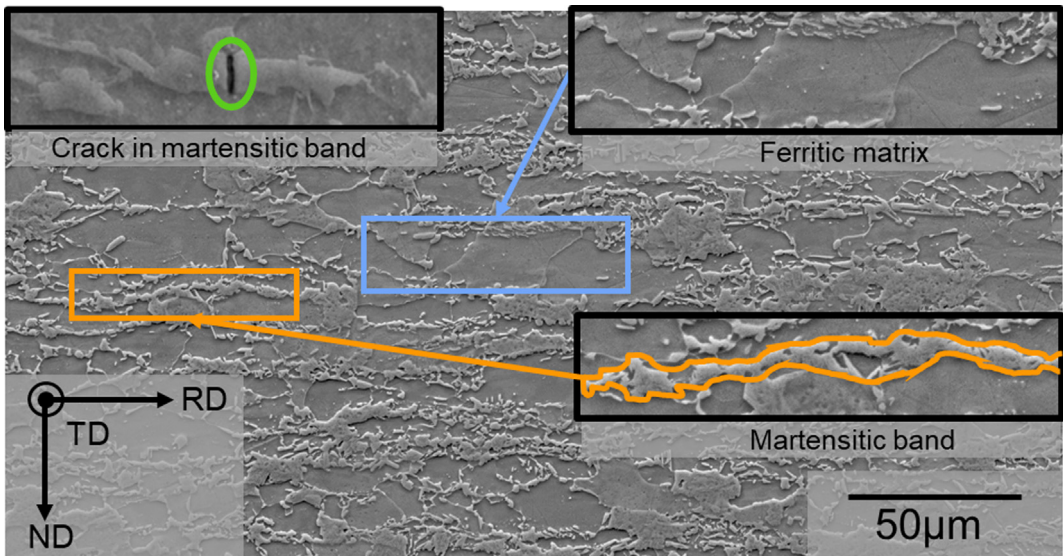


Fig. 1. Scanning electron microscopy image of the dual-phase steel DP800 microstructure acquired in the sheet plane. Examples of a martensite band, ferrite matrix and damage site (martensite cracking) from a strained sample are highlighted. RD, TD, ND refer to the rolling, transverse and normal directions.

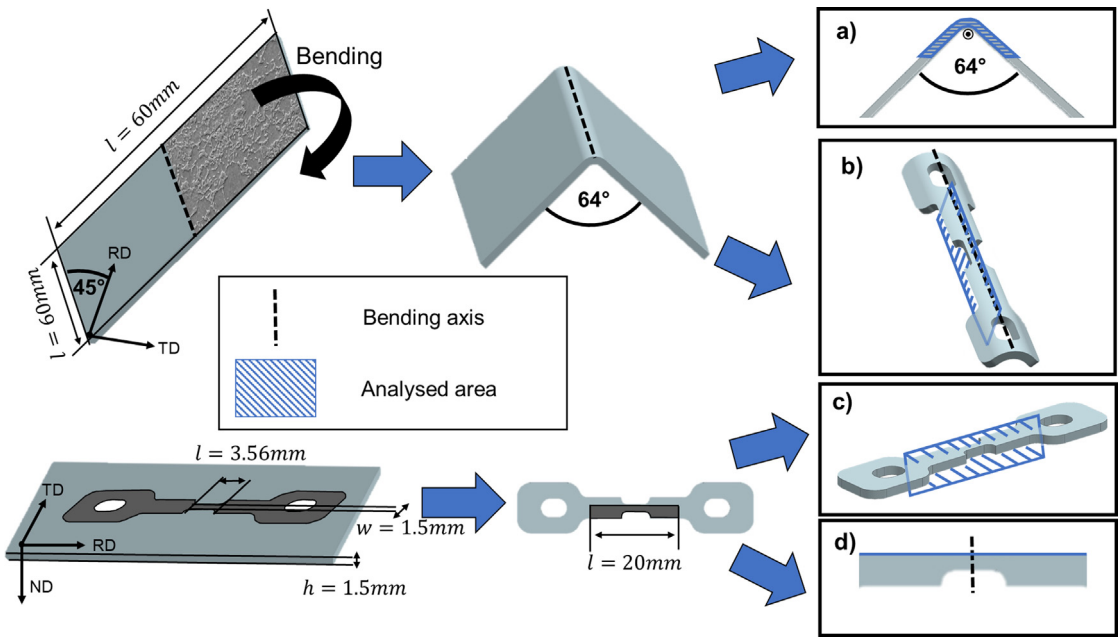


Fig. 2. Overview of the different sample geometries used in the mechanical tests to investigate the damage behaviour. a) bending test specimen b) tensile test specimen obtained from a bent sheet c) tensile test specimen d) bending test specimen obtained from a tensile test specimen. The tensile tests were terminated at $\epsilon = 12.5\%$.

2.3. Tensile tests

Aside from bending, damage analysis was also carried out after simple tensile testing. For this, another sheet of the same DP800 material used for the bending tests was machined into dog-bone shaped tensile specimens with a gauge length of 3.56 mm, a height of 1.5 mm and a gauge width of 1.5 mm, as shown in Fig. 2c. These specimens were loaded in a micro-tensile stage to 12.5 % plastic engineering strain at a speed of 10 $\mu\text{m/s}$ (~ 0.003 1/s), and then analysed regarding their damage behaviour. A strain of 12.5 % was chosen, to ensure that a comparison of the damage prevalence induced through the simple tensile test and the combined strain path (section 2.4) is possible. In this case 12.5 % is adequate, because on the one hand it is large enough to have an impact on the damage prevalence (cf. Table 2), but on the other hand it is not large enough to lead to failure of the combined specimen. In total three tensile samples were analysed in this context.

2.4. Two-step deformation tests (bending $\rightarrow$ tension)

To compare the damage behaviour investigated after individual bending and tensile tests to the damage behaviour after a combination of both tests (first bending then tension), the previously described dog-bone specimens were machined from a bent sheet, as shown in Fig. 2b, and then deformed in tension up to 12.5 % engineering strain. FE simulations showed that in all parts of the sample a positive stress triaxiality prevailed during tensile loading (shown later in Fig. 9).

2.5. Two-step deformation tests (tension $\rightarrow$ bending)

In order to investigate an additional variation of a combined strain path, the sequence of the previous two-step loading in section 2.4 was reversed such that the sample was first loaded in tension and subsequently deformed in bending to a plastic equivalent strain of 0.11 in bending direction, again measured at the outer edge of the bending side. The damage analysis was then carried out in the TD direction as shown in Fig. 2d. Additionally, the damage evolution in this strain path was observed in a quasi in-situ experiment by analysing the same area of the sample surface after tension and after subsequent bending.

2.6. Nanoindentation

Nanoindentation was used to determine the micromechanical properties of martensite and ferrite, in association with the strain path. A bending sample was measured on the surface parallel to the sheet ND in 4 different areas of deformation. This includes (i) the tensile region of the bending zone, (ii) the compression region of the bending zone, (iii) the centre region of the sample and (iv) an undeformed area. In every area, a rapid 100×100 array indentation mapping with a spacing of 700 nm between indents and a target load of 1 mN, reaching indentation depths of 150 nm in the softer ferrite phase, was conducted using a load-controlled iNano Nanoindenter (KLA, Oak Ridge, Tennessee) with the NanoBlitz 3D property mapping method. This method enables fast mapping of key values from nanoindentation experiments with a high spatial resolution, so that mappings of nano-hardness and indentation modulus are obtained with a frequency of up to 1 indent per second. The nano-hardness and indentation modulus values were obtained by evaluating the results with the Oliver and Pharr method [34,35]. Afterwards the measured hardness data of every mapping was discretised into 3 parts: The (i) ferrite and (ii) martensite phases and (iii) the interface between them. This interface is not meant to represent the properties of a physical interface, but the area, in which the plastic zone of the indents has, to all probability, stretched over both phases. This was implemented to clear the ferrite and martensite clusters of uncertain data, showing influences from both phases. In order to make the discretisation automatic a k-means clustering algorithm was applied. The three resulting clusters were then used to determine the individual phase properties at different parts of the bending sample. This procedure is exemplified for the area in the middle of the bending sample in Fig. 3 showing how the clusters are determined from a heatmap of hardness data.

2.7. Sample preparation

To detect the generated damage sites in the deformed microstructures, all of the described samples, except the nanoindentation sample, underwent the same metallographic preparation procedure. This consisted of mechanical grinding and polishing of the sample surface of interest shown in Fig. 2 with a diamond sus-

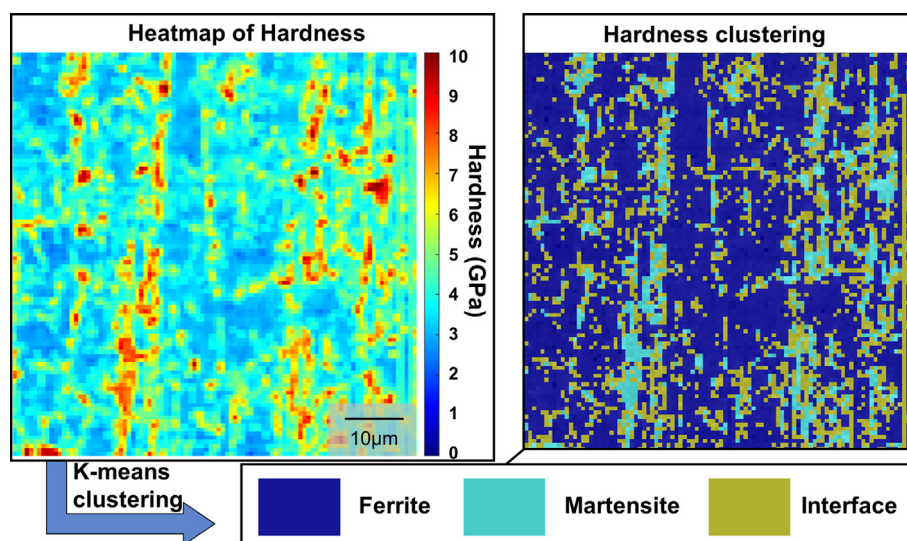


Fig. 3. Example of a nano-hardness heatmap obtained from the centre region of a bending sample (left) and a hardness clustering map from the same area obtained by using a k-means clustering algorithm (right).

pension up to 1 μm , followed by final polishing with an OPS solution up to 0.25 μm . For samples analysed in the SEM, additional etching in 1 % Nital solution for 5 s was applied. It is important to notice that all samples are only mechanically prepared once, to achieve the same surface conditions for all samples. This means that in the combined strain paths, samples were only prepared after the last deformation. The nanoindentation sample was mechanically ground and polished, but analysed in an unetched state to ensure a minimal roughness of the surface.

2.8. Microscopic damage analysis

After sample preparation, the automated identification and quantification of damage sites in the sample was conducted using panoramic imaging in a Zeiss Leo 1530 FEG-SEM (Carl Zeiss Microscopy GmbH, Oberkochen, Germany) scanning electron microscope using a secondary electron (SE) detector. Individual images in a single panoramic image had a horizontal field width of 100 μm and a resolution of 3072x2048 pixels, resulting in a lateral resolution of 32 nm/px. The panoramic images were post-processed in MATLAB 2020b to achieve similar brightness and contrast settings, by equalizing the greyscale histogram of the individual images, before they were stitched using the microscopy image stitching tool (MIST) plugin in ImageJ [36,37]. Afterwards, the images were analysed with the help of a convolutional neural network, which had already been trained and used for damage site quantification by detection and classification on the same DP800 steel and is described in more detail in references [21,24].

2.9. Finite element simulations

In addition to the experimental part of loading along different strain paths and corresponding quantitative damage analysis, finite element simulations of the strain distributions during simple bending and subsequent tensile loading were carried out. The simulation was conducted implicitly in Abaqus (Dassault Systems, 2017). For this simulation, isotropic hardening and a value of zero for friction was assumed, since it has negligible influence on the stress state of the sample. Elements of the type C3D8R were used with an edge length of 0.2 mm. The flow curve was extrapolated using the Ludwik extrapolation [38] and the corresponding formula $k_f(PEEQ) = k_f(0) + C * PEEQ^n$ with the work-hardening parameters of $k_f(0) = 518.1$, $C = 954.63$ and $n = 0.37$. For a quantitative description of the triaxiality after a strain path change, kinematic hardening must be considered, as cross-hardening effects will influence the yield characteristics of the material, which may effectively lead to different triaxialities [28]. However, in this work, we chose to use a simple work-hardening model as we only consider qualitative changes in triaxiality in our assessment of damage evolution. Also, with the intention to decrease computation time, a plane of symmetry was added in the centre of the sample (cf. Fig. 9), which included the boundary conditions of no displacement in x direction and no rotation around the y and z axes. Here, the bending test was modelled first before it was then subjected to a uniform tensile load. The results included a distribution of the plastic equivalent strain in the sample at different time steps along with the stress triaxiality and Lode angle parameter over the plastic equivalent strain in bending direction in different areas of the sample (cf. section 3.7). Theoretical studies have shown that the influence of the Lode parameter mainly affects the void shape instead of the void size or number. In later stages of deformation, it also influences the stress for void coalescence that ultimately leads to failure [39–41]. Hence, even though the Lode angle parameter was calculated, the focus of the current study was on the stress triaxiality.

3. Results

3.1. Single strain path: Bending

Three samples were taken from the described bent sheets and analysed with respect to their damage behaviour. For these analyses, the height of the panoramic image was set to be parallel to the sheet ND (cf. Fig. 2a) and the width of the panoramic image was chosen to be 1 mm. The results of this analysis for one sample are visually represented in Fig. 4. Each square represents one detected damage site. The squares are differently coloured according to the assigned mechanism, which is given in Fig. 4. Blue squares indicate voids originating from pre-existing inclusions in the microstructure (hereafter referred to as inclusion-induced voids), red squares indicate martensite cracking, green squares indicate martensite-ferrite interface decohesion and yellow squares indicate a category called notch. It is assigned to damage sites that are too small to be associated clearly with the other mentioned damage initiation mechanisms, but seem to be dominated by the stress concentration in a notch of one or two martensite islands. This category was first introduced in [21] and was shown to increase the classification accuracy for all detected voids. A damage distribution histogram, showing the number of damage sites in a certain region along the sample thickness normalized to the total number of damage sites in the measured area on the sample, is also shown in Fig. 4. Because the stress state which the sample experiences during the bending procedure changes along the sheet thickness, the sample was divided into thirds along this axis, each of which was exposed to a different stress state during bending. The top area experienced predominately tensile stresses and is shown in this work at the top of the panoramic images. The bottom area experienced mainly compressive stresses and analogously it is shown at the bottom of the panoramic images. The centre area experiences both tensile and compressive stresses and encompasses the neutral fibre. The resulting strain in this area is lower compared to the top and bottom part. The respective void area fraction (VAF), given in %, and the number of voids (NoV), normalized to 10^3 voids per mm^2 , are summarized for the tensile sides of all pure bending samples in Table 3.

The predominant damage mechanisms observed in the bent samples were identified as martensite cracking and interface decohesion. They showed a similar distribution to what is described in the literature with respect to martensite cracking being the dominant mechanism [13,21]. However, the distribution of damage initiation mechanisms is not in the scope of this work and we will focus on the damage distribution of all ductile damage sites in this work. The damage behaviour in terms of the number and area of damage sites showed considerable differences between the tensile side and the compressive side, with the latter having a smaller number of detected damage sites and a smaller void area fraction.

3.2. Single strain path: Tension

Similar to the procedure with the bending samples, three tensile samples deformed to 12.5 % plastic engineering strain were analysed with respect to their total void area fraction and number of voids. Microscopic images were obtained from the mid-section of the RD-ND plane and span the complete sample thickness (cf. Fig. 2d). As expected all sample regions (top, middle and bottom) experienced tensile stresses. The results of damage analysis for one tensile sample are shown in Fig. 5. Analogously to the bending sample, each square represents one damage site and the assigned colours indicate the respective nucleation mechanism. A normalized damage site distribution histogram through the sample thickness is also shown in Fig. 5. The statistical results for all tensile test samples are summarized in Table 4. On average, the damage distri-

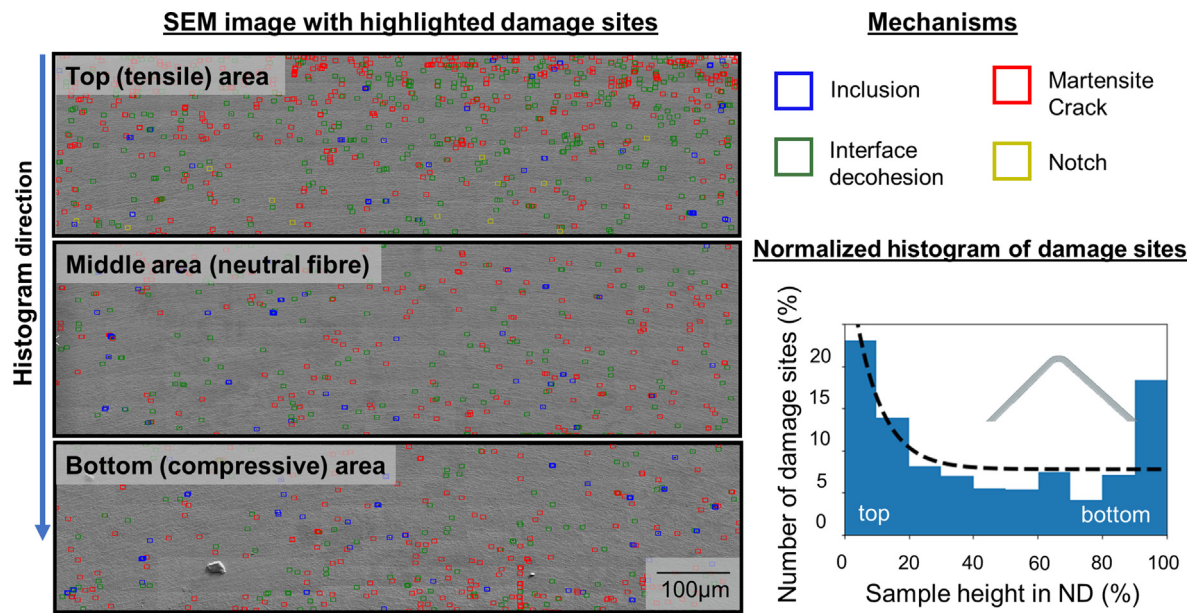


Fig. 4. SE image of detected damage sites, their initiation mechanism and distribution through the sample thickness ($\parallel$ ND) for the case of simple bending of the DP800 sheet material.

Table 3

Void area fraction and number of damage sites/voids (NoV) in the tensile side of pure bending samples.

Sample name	Bending 1	Bending 2	Bending 3	mean
Void area fraction (%)	0.035	0.021	0.027	0.028 ± 0.007
NoV ($10^3/\text{mm}^2$)	2.02	1.06	1.27	1.45 ± 0.51

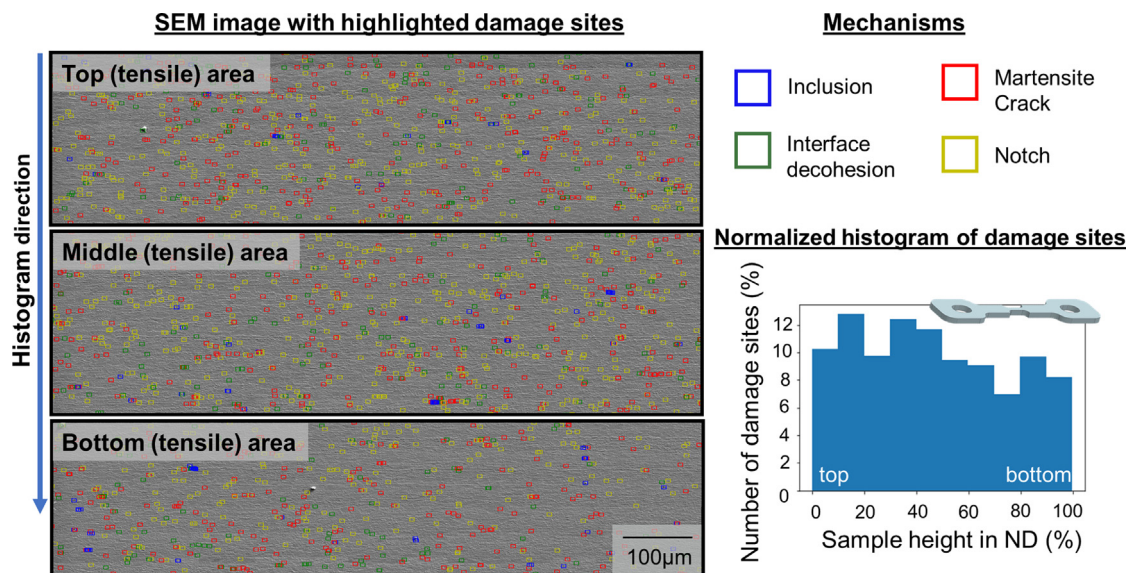


Fig. 5. SE image of detected damage sites, their initiation mechanism and distribution through the sample thickness ($\parallel$ ND) for the case of simple tension of the DP800 sheet material.

Table 4

Void area fraction and number of damage sites in pure tensile samples at 12.5 % plastic engineering strain.

Sample name	Tensile 1	Tensile 2	Tensile 3	mean
Void area fraction (%)	0.020	0.017	0.020	0.019 ± 0.002
NoV ($10^3/\text{mm}^2$)	1.43	1.23	1.53	1.39 ± 0.16

bution appears to be independent of the position in the microstructure along the normal direction. Similar to the case of the bending samples and to what is described in literature, the most prevalent damage mechanism during tension was martensite cracking [13,21].

3.3. Combined strain path: Bending followed by tension

The combined strain path applied here featured two deformation steps: 3-point bending with the bending axis at a 45° angle between RD and TD, followed by tension in the same direction. This setting was chosen to limit anisotropy effects arising from the sharp RD alignment of the martensite bands. Damage analysis of these samples was carried out analogously to the other bent and tensile-loaded samples. The complete surface of the samples was analysed in a way that the sheet ND was parallel to the vertical axis of the panoramic image (cf. Fig. 2b) in order to include the different deformation areas top, middle and bottom, stemming from the bending test. The results of 2 analysed samples are given in Table 5.

Fig. 6 shows an analysed panoramic image of a bent and subsequently tensile-loaded sample. In contrast to the individual tensile tests, the number of damage sites in this case was higher across the top (tensile) area of the sample. Moving towards the compression area, the number of damage sites decreased gradually down to a minimum value at the bottom of the sample, associated with the highest compressive strains during bending.

3.4. Combined strain path: Tension followed by bending

The second combined strain path represented a reversed case to the previous strain path, i.e. samples deformed in tensile mode

Table 5

Void area fraction and number of damage sites for combined strain path (bending → tension).

Viewing direction	Tensile direction in 45° angle between RD and TD		
Area in the bending plane	Top	Middle	Bottom
Void area fraction (%)	0.079 ± 0.007	0.039 ± 0.004	0.015 ± 0.001
NoV (10 ³ /mm ²)	5.29 ± 0.23	2.60 ± 0.25	1.22 ± 0.13

were subsequently deformed in a 3-point bending test. The damage analysis of these samples was carried out as described for the other strain paths. Here, the complete surface of the sample (again with the sheet ND being oriented parallel to the vertical axis of the panoramic image) was once more analysed in order to include the different deformation areas. In analogy to the previous case, Table 6 presents a statistical analysis of the detected damage sites arising from tensile-loading and bending.

For an easier comparison of the distributions of the number of damage sites along the ND for the investigated loading conditions, Fig. 7 presents all histograms described earlier in addition to the histogram of the sample from the combined strain path (tension → bending). The here shown distributions of normalized number of damage sites correlate closely with the void area fraction, as given in the Supplementary Material in Fig S1.

3.5. Combined strain path: The damage behaviour in the RD-TD plane

Previous investigations on damage on the same steel were conducted in the rolling plane (RD-TD plane) [21,24], rather than the RD-ND plane, as conducted here. However, to ensure that the findings from the combined strain paths are not dependent on the selected viewing direction, an additional sample from the strain path (bending → tension) was analysed in the rolling plane at different depths with respect to the ND as illustrated in Fig. 8a. One advantage of this method is that although only damaging behaviour of a few stress states can be observed, the statistics of damage sites per stress state obtained from several sections of the rolling plane is much higher than what is obtained from the RD-ND plane, because a full panoramic image can be collected from a sample surface after every cut.

Table 6

Void area fraction and number of damage sites for combined strain path (tension → bending).

Viewing direction	TD		
Area in the bending plane	Top	Middle	Bottom
Void area fraction (%)	0.142	0.074	0.006
NoV (10 ³ /mm ²)	6.37	4.00	0.23

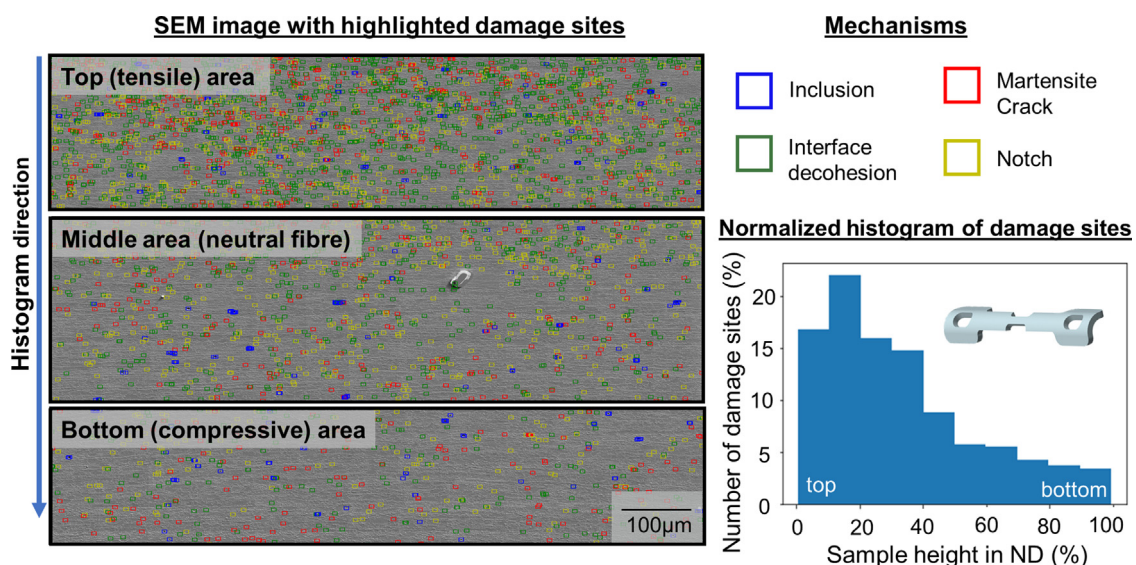


Fig. 6. SE cross-sectional panoramic image after two-step loading in bending and subsequent tension up to 12 % of the DP800 sheet material. The detected damage sites are indicated by squares coloured according to the underlying initiation mechanism and their distribution plotted along the sample height (|| ND).

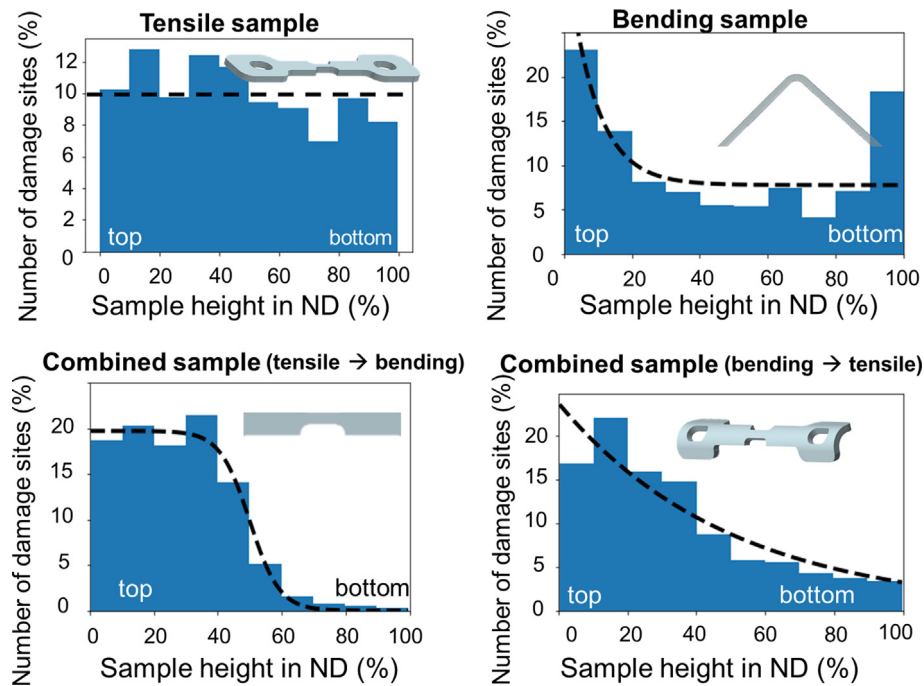


Fig. 7. Comparison of the damage behaviour in terms of the distribution of the number of damage sites along the ND in deformed DP800 samples deformed along different strain paths (simple tension, simple bending, two-step loading (tensile → bending) and two-step loading (bending → tensile). Schematic fits to the shown distributions are included to indicate the average damage behaviour in each case (black dashed lines).

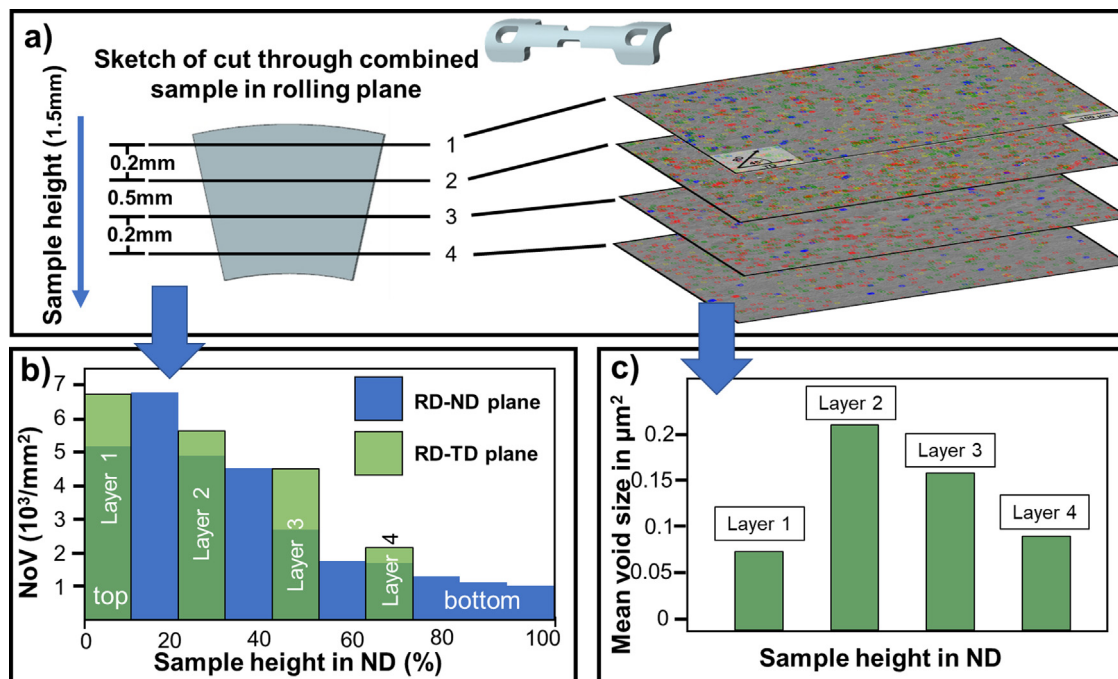


Fig. 8. Damage analysis in the RD-TD plane of a sample of combined strain path (bending → tensile) (a) schematic illustration and analysed SEM panoramic images of the investigated samples at different depths of the rolling plane. (b) NoV along the sample height determined in plan (green) and side view (blue as in Fig. 6) and (c) mean void area at different positions of the rolling plane with respect to ND. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The results of this analysis reveal the same trend regarding the number of voids (cf. Fig. 7), i.e. a decreasing number of voids from the tensile area to the compressive area (Fig. 8b). Furthermore, it becomes apparent that the normalized number of damage sites

can be directly and quantitatively compared between the RD-TD plane (highlighted in green) and the RD-ND plane (highlighted in blue), used for the analysis of damage on the rest of the samples. This finding suggests that for similar conditions, a direct compar-

ison with results obtained in literature from a RD-ND plane is feasible. A peak in the mean individual void size around the centre (Fig. 8c) could also be observed.

3.6. Nanoindentation

From the nanoindentation measurements, the hardness of ferrite, martensite and their interfacial area was determined in an undeformed sample and for the tensile, centre and compression area of a bent sample, as explained earlier in section 2.6. Here, we do not set out to resolve the microstructural constituents spatially but use this technique to gather a large dataset of nano-hardness & indentation modulus values to receive a high statistical relevance. The results are summarized in Table 7.

3.7. Finite element simulations

With the help of the finite element simulations, a distribution of the local plastic equivalent strain in bending direction was calculated for bending and the combined strain path (bending → tension). Additionally, the triaxiality was determined for every point in the sample and then plotted against the plastic equivalent strain in bending direction to obtain the change of

Table 7
Hardness of different phases and the interfacial area measured by nanoindentation on a cross-section in differently strained areas of a bent sample.

	Hardness ferrite (GPa)	Hardness martensite (GPa)	Hardness interface (GPa)
Area 1 (undeformed)	3.47 ± 0.31	7.88 ± 0.95	4.98 ± 0.40
Area 2 (tensile area)	4.17 ± 0.32	6.89 ± 0.63	5.1 ± 0.32
Area 3 (compressive area)	4.13 ± 0.33	6.69 ± 0.62	4.98 ± 0.32
Area 4 (centre area)	3.81 ± 0.34	7.34 ± 0.75	5.22 ± 0.44

stress state over deformation for the different areas of the sample. The obtained data for the centre of the sample, the edge of the tensile side and the edge of the compression side, as well as the PEEQ distribution after bending and after the combined strain path (bending → tension) are shown in Fig. 9.

By considering the onset of necking in the tensile sample and comparing it to the necking in the simulated sample, a local maximal PEEQ was calculated for the individual areas, which allows us to plot the change in triaxiality up to the final deformation step. On the tensile side, the stress triaxiality remains positive during the complete forming process. Yet, once the loading is changed from bending to tensile, the triaxiality decreases from 0.57 to 0.30. The centre of the sample experiences the least amount of deformation during the bending test and hence also the smallest total strain. However, during its small deformation during the bending step the evaluated element experienced a negative triaxiality. As soon as the tensile loading starts, the triaxiality turns positive and assumes a value of approximately 0.30, just as it does on the tensile side, but increases to a triaxiality of approximately 0.42. The change in triaxiality is strongest for the compressive side. Here, the sample experiences a negative triaxiality starting at −1 and increasing to −0.6 during bending deformation. Yet, as soon as the tensile loading starts, the triaxiality becomes positive and again becomes approximately 0.30 here as well. Generally speaking, it also becomes apparent that the sample still deforms homogeneously after the bending load, as the local deformation, represented by the PEEQ, during the tensile test is ~ 0.28 in all three areas. The impression of homogenous deformation is underlined visually by the similar strain distribution of the combined sample in the orange box in Fig. 9 compared to the distribution after bending shown in the blue box.

4. Discussion

4.1. Microscopic damage behaviour under positive stress triaxiality

The microstructural parameters chosen in this study to quantify microscopic damage in the deformed samples are the Number of Voids (NoV) normalized to 10^3 voids per mm^2 and the Void Area

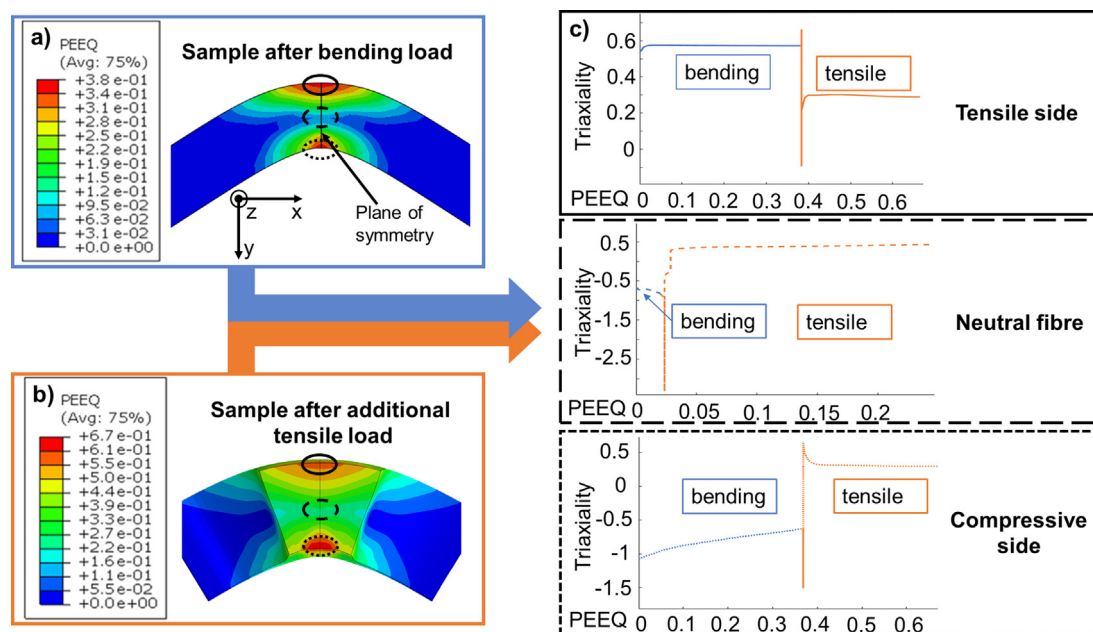


Fig. 9. (a) PEEQ distribution after bending with highlighted plane of symmetry, (b) after combined strain path (bending → tension) and (c) the corresponding simulated triaxiality as a function of PEEQ in bending direction for different regions of the sample.

Fraction (VAF) in %. Both of these parameters are simultaneously analysed and compared to reveal differences in the nucleation and growth of voids between different strain paths. In addition to the global parameters NoV and VAF, local information of individual voids were also considered for an assessment of individual void size as a function of different stress states.

There are three areas in the deformed samples exhibiting positive triaxiality: The entire tensile sample, the tensile side of the bending sample and the centre of the sample subjected to the combined strain path (bending → tension). Focusing on the local damage distribution in the tensile samples, we observe that no change in damage density is apparent when moving along the normal direction of the sheet (cf. Fig. 5). This can be expected for uniaxial tension, as the stress state does not change significantly over the thickness of the sample. The homogenous distribution of positive triaxiality in this case results in a mean VAF of 0.019 ± 0.002 % and a NoV of $1.39 \pm 0.16 \times \frac{10^3}{\text{mm}^2}$.

In contrast to the tensile tests, the bending samples show a clear trend of decreasing NoV and VAF along ND (Fig. 4), which is attributed to the changing stress state in this direction. The positive triaxiality of 0.57 at the edge of the tensile side (Fig. 9) leads to a VAF of 0.028 ± 0.007 % and a NoV of $1.45 \pm 0.51 \times \frac{10^3}{\text{mm}^2}$. It also gives rise to larger voids with a mean size of 0.195 ± 0.019 μm^2 . This observation is in good agreement with the literature [25,42]. It is important to highlight that the error given for the void sizes in the current work has been determined by comparing the mean void size of different samples. Because the void size distribution spans over a wide range of void sizes, an error calculated by the standard deviation of the distribution results in a larger error than calculating the standard deviation of the mean void size of different samples.

The centre area (neutral fibre) in the combined strain path (bending → tension) was virtually only subjected to positive triaxiality, because the initial bending deformation induces only a minimal amount of strain (~ 0.02 , cf. Fig. 9). This gave rise to slightly higher NoV of $2.60 \pm 0.25 \times \frac{10^3}{\text{mm}^2}$ and VAF of 0.039 ± 0.004 %, since the centre region was also affected by the stress-state in the tensile region that facilitated damage nucleation and growth. Additionally, it was observed that the neutral fibre shifted towards the compression zone by ~ 60 μm (Fig. 9).

4.2. Microscopic damage behaviour under negative stress triaxiality

Negative triaxialities are generally not known to contribute with void nucleation or void growth. According to Bao and Wierzbicki [43], a triaxiality lower than $-1/3$ will not even lead to fracture of the sample, as there is no damage accumulation taking place. This trend can indeed be observed in the compressive region of the bending samples. It is shown that the NoV declines and stays roughly constant once the middle of the sample is reached and only compressive stresses are present. The only exception for this is the edge of the sample, which shows a strong increase in number of damage sites. However, this increase is related to surface artefacts introduced to the sample surface during the grinding step and is not related to damage formation. Still, it is important to distinguish between two areas with compressive stresses: (i) the area near the centre and (ii) the area near the edge of the compressive side. Although neither of these areas show significant damage nucleation or growth, the latter experienced more deformation than the centre area of the bending sample. The different amount of stress experienced by these two areas can be seen in Fig. 9. This is suggested to have an impact on the mechanical properties of the material due to strain hardening. As larger deformation leads to more strain hardening, the microstructure in the compression zone is believed to experience larger strain hardening than the counter-

part area around the centre. This was corroborated via nanoindentation. The trend which the results show, increasing from a hardness of 3.81 ± 0.34 GPa in the ferrite matrix in the centre of the sample, to a hardness of 4.13 ± 0.33 GPa in the deformed ferrite in the compression zone, meets the expectations for strain hardening.

4.3. Microscopic damage behaviour after two-step deformation

4.3.1. Negative triaxiality + positive triaxiality

Damage evolution after a strain path change is more complex than damage evolution under simple strain paths. The difference between the effects on the damage distribution in the samples, can be observed in the combined strain paths. Here, a deformation with a negative triaxiality followed by a deformation with a positive triaxiality can be found in the compression zone of the sample of complex strain path (bending → tension). As established above, deformation with negative triaxiality does not induce damage in the form of void formation. Hence the NoV and VAF after the complete strain path are expected to be equal or lower than in the tensile tests. This expectation is met as this area assumed a NoV of $1.22 \pm 0.13 \times \frac{10^3}{\text{mm}^2}$ and a VAF of 0.015 ± 0.001 %, which indicates less damage than in the tensile samples. This is visualized in Fig. 10. Through a loading with negative triaxiality before the tensile test, the VAF could be reduced by 21 % and the NoV by 12 % in comparison to the simple tensile test.

The lower amount of damage in the compression area of the bending → tension combined specimen is attributed to a strain hardening effect. It is already established that damage formation in DP steels originates from the heterogeneous plastic deformation of martensite and ferrite, as the ferrite is softer and more ductile than the martensite as shown in Table 7. Yet, as was shown with the help of nanoindentation, the ferrite phase experiences strain hardening during plastic deformation. In compression, this proceeds in the absence of void formation, and thereby only the mechanical contrast, in particular the difference of nano-hardness, between the two phases is reduced. In this regard, Tian et al. [33] reported that the strain hardening behaviour and hardness of the ferrite phase are important to understanding the damage formation in dual-phase steels, as microstructures with a less pronounced mechanical contrast show reduced damage formation compared to those with a high mechanical contrast. This agrees fairly well with the observed strain hardening of ferrite and the exponential decrease of damage sites when moving from the neutral fibre towards the compression zone (cf. Fig. 6).

Furthermore, by comparing the mean void size from the centre area of a sample of combined strain path (bending → tension) (0.149 ± 0.001 μm^2) with the mean void size in the compression zone of the same sample (0.130 ± 0.005 μm^2), it can be observed that the growth of voids in the deformed microstructure is less pronounced relative to what was seen as they do in the undeformed microstructure. This also agrees well with the aforementioned hypothesis that strain hardening hinders not only the nucleation of new voids but also the growth of existing ones.

Even on a macroscopic scale, compression prior to tension leads to larger fracture strains, which has already been macroscopically observed by other researchers [29,44]. By combining a statistical damage and indentation analysis we can show directly, that through prior plastic deformation with a negative triaxiality, potential damage formation during following forming steps can be decreased. Linking the important result of a higher fracture strain of the material revealed through macroscopic compression-tension tests, to the reduced damage formation observed in this work, the larger fracture strains can be explained by the reduced damage prevalence.

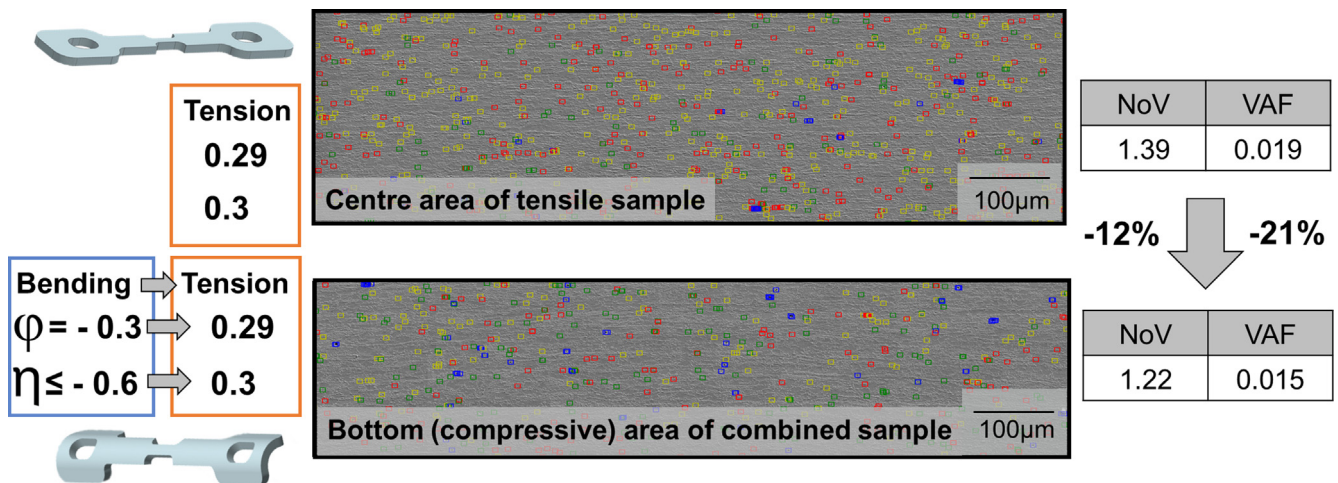


Fig. 10. Comparison of the microstructure and damage parameters between the centre area of a tensile sample and the compression area of a sample from combined strain path (bending → tension). The decrease of damage in the latter reveals the positive role loading with negative triaxiality in a two-step process.

4.3.2. Positive triaxiality + positive triaxiality

The tensile side of the sample of the combined strain path (bending → tension) experienced positive triaxialities in both deformation steps. During bending a triaxiality of 0.57 persisted until a maximum PEEQ of 0.38 at the outer edge of the tensile side, which was then reduced to 0.30 during subsequent tension up to a total maximum strain of 0.67. This led to larger damage parameters, i.e. NoV of $5.29 \pm 0.23 \times 10^3 / \text{mm}^2$ and a VAF of $0.079 \pm 0.007 \%$, as compared to the previous case of the compression side. Because the total imposed strain was larger in this combined sample than in the individual tensile and bending tests, larger NoV and VAF values are reasonable. This trend is in good agreement with an earlier work by Kusche et al. who carried out a systematic investigation of the influence of strain and triaxiality on the evolution of deformation-induced damage in DP800 [25]. They concluded that on a global level, the applied plastic strain has a sizeable influence on the nucleation of new voids, while the stress triaxiality is the most important factor related to the extent of void growth, even where locally in a dual phase microstructure the plastic strain and triaxiality may vary considerably. This was indeed observed in the current experiments as well.

Considering the estimated mean void size of $0.150 \pm 0.020 \mu\text{m}^2$ on the tensile side of the combined strain path (bending → tension), it can be concluded that void growth is not the main reason for the increase of the VAF at this point of the strain path combination. In fact, after the second deformation step in tension, the mean void size in the tensile area of the bending sample decreases. This behaviour can only result from a high void nucleation rate and limited growth, since void closure is not expected for positive triaxialities. In this regard, limited void growth can be explained by the decrease in triaxiality (0.57 → 0.30) during tension, rendering void nucleation the primary driver for the increase of the VAF. Another aspect for limited void growth is that in the second step of tension, the principle loading direction is rotated by 90° . This means that microstructural sites, which were not prone to void formation in the first loading direction can facilitate void nucleation in a different loading direction. As a result, nucleation of new voids may become more favourable than growth of pre-existing voids in a strain hardened ferrite matrix.

The second possibility to impose two positive triaxialities considered in this work is a bending test conducted subsequently to a tensile deformation. In this case, the same trend of exponential

damage increase was evident in terms of a NoV of $6.37 \times \frac{10^3}{\text{mm}^2}$ a VAF of 0.142 % and a mean void size of $0.220 \mu\text{m}^2$ for this sample. However, it is important to bear in mind that the quantified damage in this case cannot be directly compared to the previous case of bending → tension. This is because (i) the strain levels during bending in the combined strain path bending → tension are higher than in the combined strain path tension → bending, (ii) the stress-state varies in the two bending cases due to different sample geometries, and (iii) the loading direction was not changed in the combined strain path tension → bending. Despite these differences, the results again show a clear trend of a strong increase in both damage number and area when combining two strain paths with positive triaxiality. By comparing the mean void sizes, it becomes clear that the void size is larger for the strain path (tension → bending). This can be explained by the fact that in contrast to the strain path (bending → tension), the deformation mode with a higher triaxiality was carried out last and the loading direction was not changed, rendering void growth more favourable.

Even if the influence of the sample geometry is unclear in this case, we therefore suggest that a change in loading direction during forming may be favourable for reducing damage, since as shown here all damage parameters were lower for a sample with a change of loading direction, even though it experienced a higher amount of strain. In Fig. 11, the microstructures and evolution of damage parameters are shown for both strain paths in a direct comparison.

4.3.3. Positive triaxiality + negative triaxiality

The compression side of the combined strain path (tension → bending) represents the case, where deformation proceeded under positive triaxiality in the tensile test and was then changed to negative triaxiality during subsequent bending. In contrast to all other combinations discussed so far, this combination is the only one that actually reduces the damage parameters through further deformation. The VAF of $0.019 \pm 0.002 \%$ and NoV of $1.39 \pm 0.16 \times \frac{10^3}{\text{mm}^2}$ measured in the tensile test samples are reduced to 0.006 % and $0.23 \times \frac{10^3}{\text{mm}^2}$ respectively in this case. Because the sample experiences compressive stresses on this side, voids, nucleated during tension, abate, thereby decreasing the VAF. To visualize the effect of compressive stresses on the shape and size of voids, bending of the combined strain path (tension → bending) was performed in a quasi in-situ experiment.

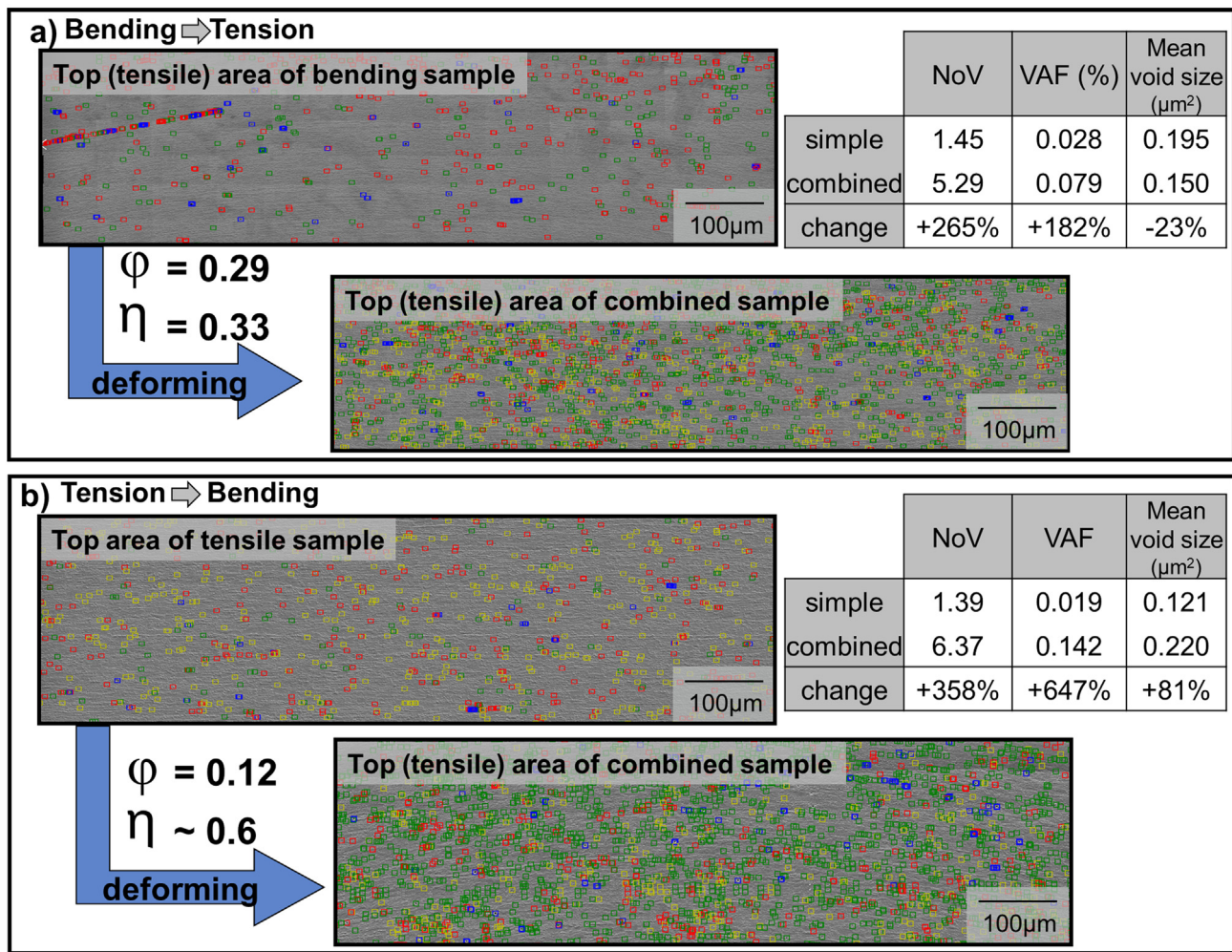


Fig. 11. Microstructure and damage parameters of top area of a bending sample, top area of the sample from the combined strain path (bending $\rightarrow$ tension), top area of a tensile test sample and the top area of combined sample from the combined strain path (tension $\rightarrow$ bending) visualizing the damage evolution for both combined strain paths.

Fig. 12 shows the microstructure in the compression zone of a sample of the combined strain path (tension $\rightarrow$ bending) before and after the bending step. Three different voids are highlighted by differently coloured boxes showing that the plastic deformation can lead to a change of void shape, a reduction of void area and even to void closure.

Presumably, the area of the void undergoing void closure is decreased to the extent that it cannot be reliably detected by SEM any longer, when using the here applied resolution, and hence the NoV is also decreased in this area. This behaviour is also evident in terms of the in the mean individual void area of $0.110 \mu\text{m}^2$ in this region, which is slightly smaller than its counterpart in the tensile tests. The total decrease of microstructural damage sites under further deformation with negative triaxiality is visualized in Fig. 13, where a microstructure and the damage parameters after tensile deformation are shown next to the microstructure and damage parameters in the compression side of the combined strain path (tension $\rightarrow$ bending). The presumption that voids are becoming too small to detect instead of a complete healing, suggests that the NoV is possibly underestimated in this region. Whether or not a healing of damage sites is likely in this process is not clear, though it has been observed and modelled for forging and rolling processes [45–47]. These works report that this phenomenon is possible at elevated temperatures and/or large strains. It is therefore uncertain if the attained strains in the cur-

rent deformation experiment were large enough to trigger healing of damage sites. Hence it is possible that the negative influence of damage sites on the mechanical properties is still present, even if they were no longer detected in the SEM.

4.4. Influence of strain path on damage formation

As discussed in section 4.3.1 and shown in Fig. 10 we argued that a strain hardened microstructure can inhibit void nucleation and growth, and thereby reduces damage formation. This means that a material can undergo the same deformation process (e.g. uniaxial tension) but accumulates less damage sites, if it was priorly deformed under negative triaxiality in a different direction, as shown in Fig. 14 with the values from Table 4 and Table 5. Such samples are hence expected to have a better mechanical response, for example in the case of crashworthiness or longevity during cyclic loading. Another strain path-related aspect influencing damage formation is the loading direction, which can be changed in the course of combined deformation steps. Comparing both complex strain paths investigated here, suggested that a change of direction is favourable for inhibiting damage in the microstructure.

Additionally, the damage parameters of the top and bottom areas from the combined strain path (bending $\rightarrow$ tension), which experienced different stress states, i.e. positive + positive triaxiality in the tensile zone and negative + positive triaxiality in the com-

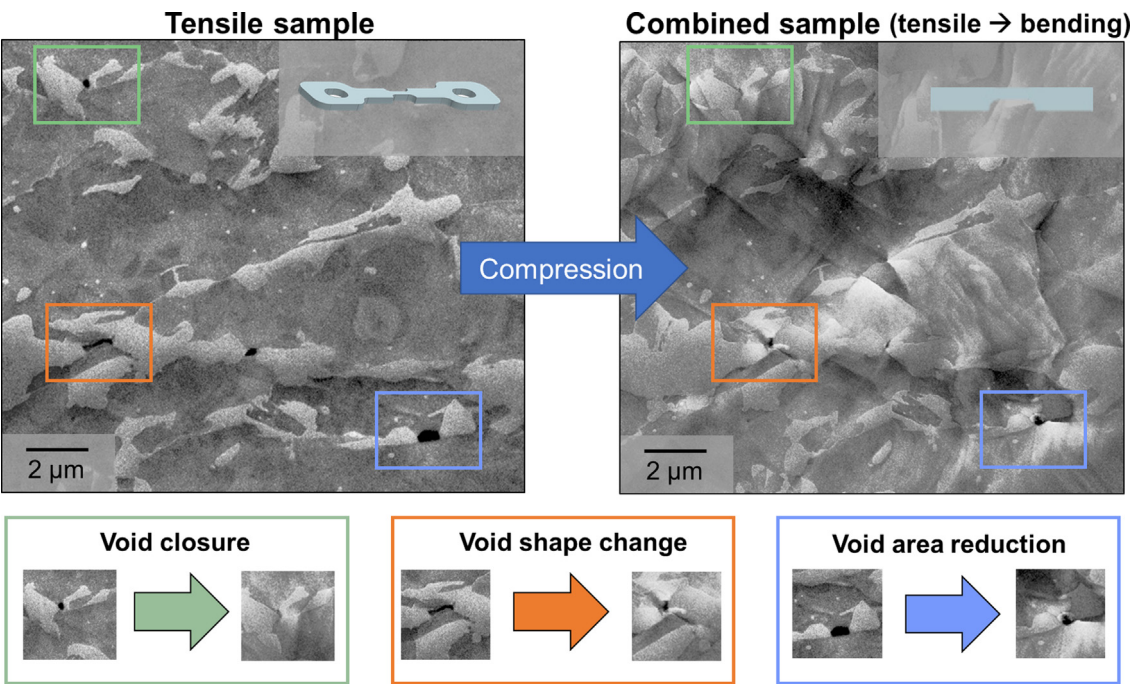


Fig. 12. In-situ observation of a, through tensile deformation pre-damaged, microstructure in the compression zone of a bending test. Three voids are highlighted as an example for the different observed effects on damage sites during compression. Green shows a void undergoing void closure, orange shows a void undergoing a change of shape and blue shows a void that is mainly reduced in area. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

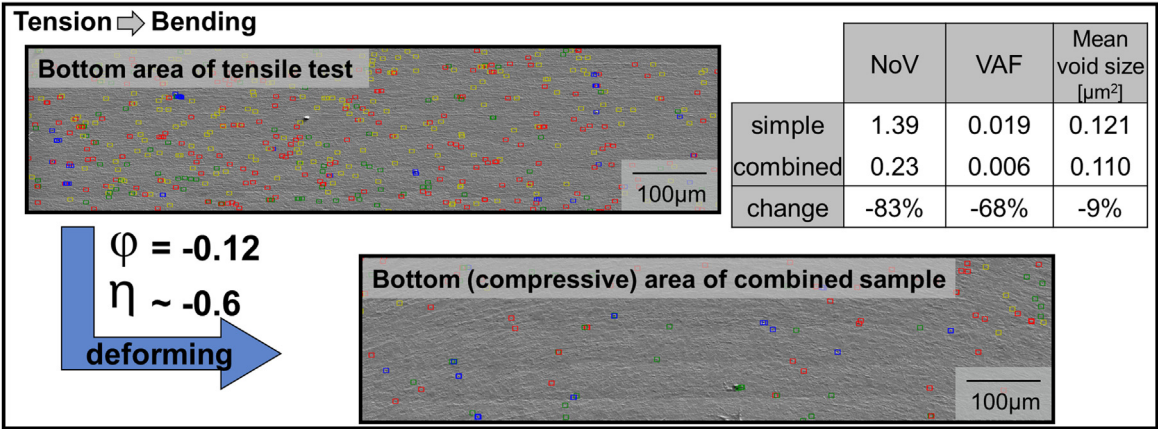


Fig. 13. Microstructure and damage parameters of bottom area of a tensile test sample and the bottom area of combined sample from strain (tension → bending) visualizing the void closure and damage decrease.

pressive zone, show that pre-existing damage also influences the further damage formation. This is established by comparing the number of newly nucleated voids in the compression area to that in the tensile area. Both the tensile and compressive area show the same amount of strain and also show the same amount of strain hardening after bending evident by the measured ferrite hardness (4.13 ± 0.33 GPa in the compression zone and 4.17 ± 0.32 GPa in the tensile zone). Accordingly, the nucleation rate of voids should decrease equally, leading to an equal number of newly nucleated voids in both areas. But, after the following tensile deformation the opposite trend is observed, i.e. the NoV in the compression area increases by $\sim 0.4 \times 10^3$ voids per mm² and in the tensile area by $\sim 3.8 \times 10^3$ voids per mm². This demonstrates that damage nucleation is more pronounced in the tensile area of the sample, where more voids were already present. Such drastic

increase in the number of damage sites has also been observed by Kusche et al. [25]. Fig. 15 shows this trend by depicting the NoV as a function of plastic equivalent strain, as was published in [25]. Based on Table 5, Fig. 15 also features the determined NoV of the of the tensile and compression area at a plastic strain of 0.125, highlighted as blue and orange triangles respectively. At a similar strain the NoV determined in [25] showed a value of $\sim 2.5 \times \frac{10^3}{\text{mm}^2}$, which lies between the NoV values determined in the current work. Additionally, a qualitative trend of the NoV over plastic strain in tensile direction, expected for a tensile test, is given as dashed lines for both samples. This comparison suggests that the damage evolution curve can be manipulated (shifted along the y-axis) depending on the imposed strain path. By extending the conducted tests to include the effect of different pre-tensile strains

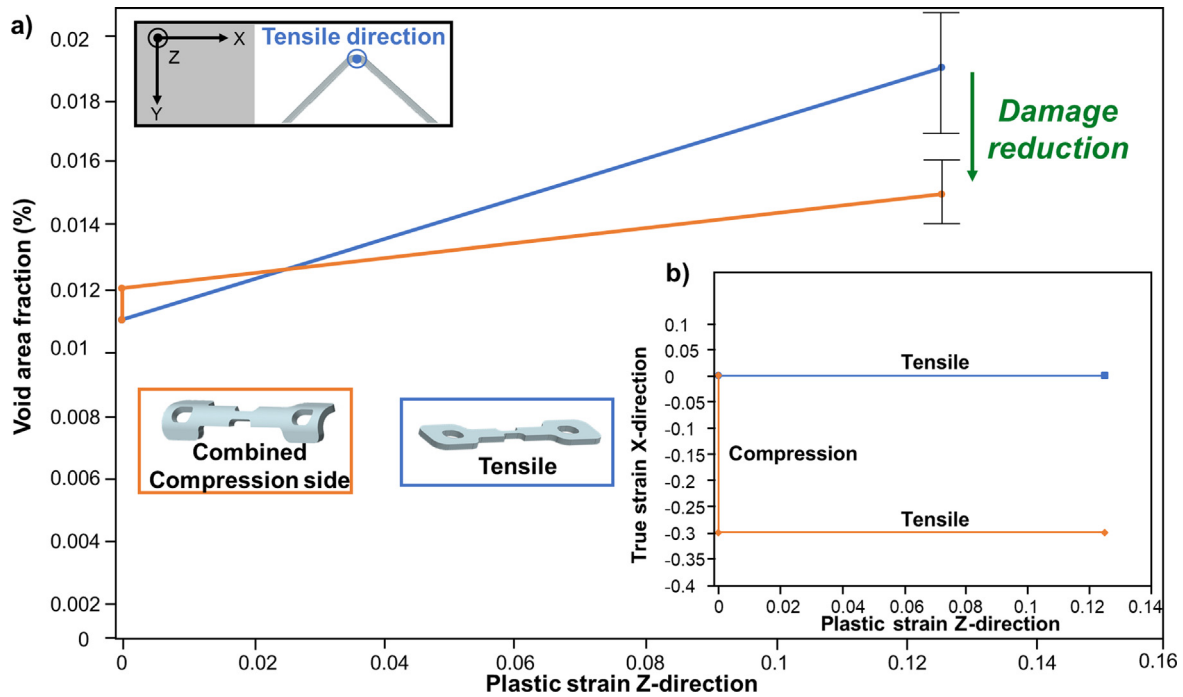


Fig. 14. Change of void area fraction with strain in tensile direction for the tensile test and the combined strain path (tension → bending) in a). The complete strain path in both directions is shown for both samples in b).

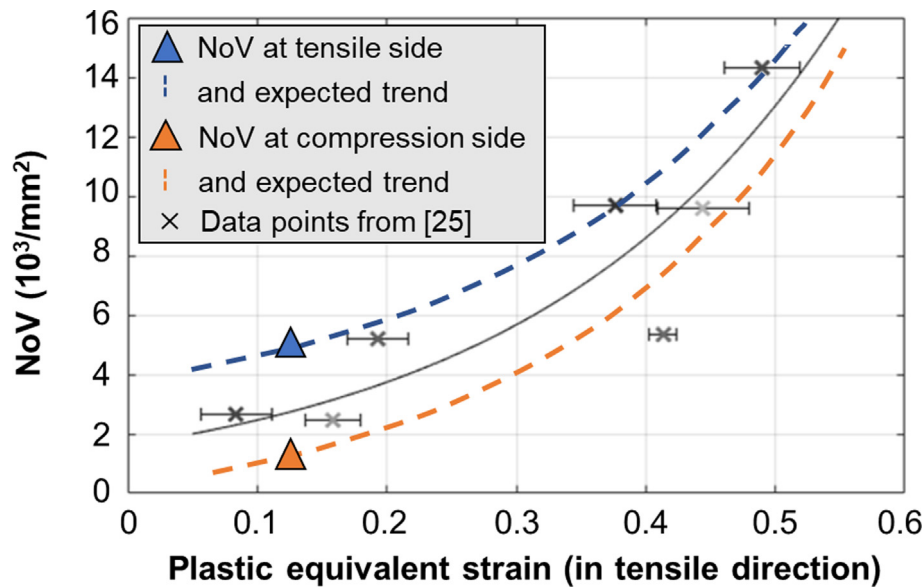


Fig. 15. Number of voids as a function of tensile strain in reproduced from Ref. [25]. The triangles correspond to data points from the current work measured in the tension and compression areas of the bent and tensile loaded sample. The corresponding dashed curves represent the expected exponential trend of damage increase in these regions with progressing tensile deformation.

and bending strains on subsequent two-step deformation the shift of this curve could be evaluated in more detail. By considering the effects of different directional changes, as well as featuring in-situ observations, the understanding of the mechanisms behind individual void growth in complex loading scenarios can be understood to a larger extent. It is also of interest whether voids that opened during a positive triaxiality and shrink during forming with a negative triaxiality, as in the compression area of the complex strain path (tension → bending), can actually be healed. Finally, determining the effect of void number and void area reduction

on the mechanical properties of the material, for example on the crashworthiness, would finalise how important the damage reduction is for the application of the material.

5. Conclusions

We studied the formation and growth of microstructural damage sites in DP800 steel subjected to different strain paths comprising simple tension, simple bending and more complex cases of

two-step loading in tension followed by bending or in bending followed by tension. For damage analysis, high-resolution SEM imaging was used in combination with an automated void analysis algorithm. This was complemented by nanoindentation mappings and FE simulations. The results showed that the interaction of combined strain paths featuring different stress states plays an important role in the global damage formation, which can also lead to strong local damage gradients in the deformed microstructures. The main conclusions are summarized as follows:

- When deformation first proceeds under a negative stress triaxiality and then, due to a strain path change, under positive triaxiality, damage formation is reduced owing to a favourable effect of strain hardening on inhibiting void nucleation and growth.
- When deformation proceeds under positive triaxiality during the first and the second strain path, damage formation is promoted, despite strain hardening, because the number of voids is already high after the first strain path.
- Changing the loading direction between the first and second strain path decreases the rate of damage formation, as compared to a fixed loading direction during both strain paths.
- In strain paths where deformation under positive triaxiality is followed by deformation under negative triaxiality, the damage parameters represented by the number and area of voids show a decrease that is likely related to a reduction of ductile damage by void closure due to negative hydrostatic pressure.
- Out of the different strain path combinations explored in this study, forming at a negative stress triaxiality prior to subsequent steps at a positive triaxiality in combination with a change in loading direction is a good strategy to decrease ductile damage.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The authors express their gratitude to the Deutsche Forschungsgemeinschaft (DFG) for financial support in context of the Collaborative Research Centre CRC/Transregio 188/2 "Damage Controlled Forming Processes", project B02, project no. 278868966.

This work is funded by the Bundesministerium für Bildung und Forschung, and the state governments participating on the basis of the resolutions of the GWK for national high performance computing at universities (www.nhr-verein.de/unsere-partner).

We would also like to extend our gratitude to Mr. Philipp Lenemann from the Institut für Umformtechnik und Leichtbau in Dortmund for conducting the FE simulations.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.matdes.2023.112016>.

References

- [1] C. Zhang, R. Wang, G. Song, Effects of pre-fatigue damage on mechanical properties of Q690 high-strength steel, *Constr. Build. Mater.* 252 (2020) 118845.
- [2] A.-P. Pierman, O. Bouaziz, T. Pardoen, P. Jacques, L. Brassart, The influence of microstructure and composition on the plastic behaviour of dual-phase steels, *Acta Materialia* 73 (2014) 298–311.
- [3] S.K. Paul, Micromechanics based modeling of Dual Phase steels: Prediction of ductility and failure modes, *Comput. Mater. Sci.* 56 (2012) 34–42.
- [4] A.E. Tekkaya, N.B. Khalifa, O. Hering, R. Meys, S. Myslicki, F. Walther, Forming-induced damage and its effects on product properties, *CIRP Ann.* 66 (1) (2017) 281–284.
- [5] S. Heibel, T. Dettinger, W. Nester, T. Clausmeyer, A.E. Tekkaya, Damage mechanisms and mechanical properties of high-strength multiphase steels, *Materials* 11 (5) (2018) 761.
- [6] G. Avramovic-Cingara, Y. Ososkov, M. Jain, D. Wilkinson, Effect of martensite distribution on damage behaviour in DP600 dual phase steels, *Materials Science & Engineering: A* 516 (1–2) (2009) 7–16.
- [7] Q. Lai, O. Bouaziz, M. Gouné, L. Brassart, M. Verdier, G. Parry, A. Perlade, Y. Bréchet, T. Pardoen, Damage and fracture of dual-phase steels: Influence of martensite volume fraction, *Materials Science & Engineering: A* 646 (2015) 322–331.
- [8] A. Bahrami, S.M. Anijdan, A. Ekrami, Prediction of mechanical properties of DP steels using neural network model, *J. Alloy. Compd.* 392 (1–2) (2005) 177–182.
- [9] A. Bag, K. Ray, E. Dwarakadasa, Influence of martensite content and morphology on tensile and impact properties of high-martensite dual-phase steels, *Metall. Mater. Trans. A* 30 (5) (1999) 1193–1202.
- [10] C.C. Tasan, J.P. Hoefnagels, M. Diehl, D. Yan, F. Roters, D. Raabe, Strain localization and damage in dual phase steels investigated by coupled in-situ deformation experiments and crystal plasticity simulations, *Int. J. Plast.* 63 (2014) 198–210.
- [11] N. Saeidi, F. Ashrafizadeh, B. Niroumand, F. Barlat, EBSD study of damage mechanisms in a high-strength ferrite-martensite dual-phase steel, *Journal of Materials Engineering Performance* 24 (1) (2015) 53–58.
- [12] J.P. Hoefnagels, C.C. Tasan, F. Maresca, F.J. Peters, V.G. Kouznetsova, Retardation of plastic instability via damage-enabled microstrain delocalization, *J. Mater. Sci.* 50 (21) (2015) 6882–6897.
- [13] J. Kadkhodapour, A. Butz, S.Z. Rad, Mechanisms of void formation during tensile testing in a commercial, dual-phase steel, *Acta Materialia* 59 (7) (2011) 2575–2588.
- [14] C. Landron, E. Maire, O. Bouaziz, J. Adrien, L. Lecarme, A. Bareggi, Validation of void growth models using X-ray microtomography characterization of damage in dual phase steels, *Acta Materialia* 59 (20) (2011) 7564–7573.
- [15] C. Landron, O. Bouaziz, E. Maire, J. Adrien, Experimental investigation of void coalescence in a dual phase steel using X-ray tomography, *Acta Materialia* 61 (18) (2013) 6821–6829.
- [16] G. Avramovic-Cingara, C.A. Saleh, M. Jain, D. Wilkinson, Void nucleation and growth in dual-phase steel 600 during uniaxial tensile testing, *Metall. Mater. Trans. A* 40 (13) (2009) 3117–3127.
- [17] H. Ghadbeigi, C. Pinna, S. Celotto, J. Yates, Local plastic strain evolution in a high strength dual-phase steel, *Materials Science & Engineering: A* 527 (18–19) (2010) 5026–5032.
- [18] D.L. Steinbrunner, D. Matlock, G. Krauss, Void formation during tensile testing of dual phase steels, *Metall. Mater. Trans. A* 19 (3) (1988) 579–589.
- [19] E. Ahmad, T. Manzoor, K.L. Ali, J. Akhter, Effect of microvoid formation on the tensile properties of dual-phase steel, *Journal of Materials Engineering Performance* 9 (3) (2000) 306–310.
- [20] C. Landron, O. Bouaziz, E. Maire, J. Adrien, Characterization and modeling of void nucleation by interface decohesion in dual phase steels, *Scripta Comput. Sci. Appl. Math. Materialia* 63 (10) (2010) 973–976.
- [21] C. Kusche, T. Reclik, M. Freund, T. Al-Samman, U. Kerzel, S. Korte-Kerzel, P. Pławiak, Large-area, high-resolution characterisation and classification of damage mechanisms in dual-phase steel using deep learning, *PLoS one* 14 (5) (2019).
- [22] T.M. Ostormujof, R.P.R. Purohit, S. Breumier, N. Gey, M. Salib, L. Germain, Deep Learning for automated phase segmentation in EBSD maps, A case study in Dual Phase steel microstructures, *Materials Characterization* 184 (2022) 111638.
- [23] W. Liu, J. Lian, S. Münstermann, Damage mechanism analysis of a high-strength dual-phase steel sheet with optimized fracture samples for various stress states and loading rates, *Eng. Fail. Anal.* 106 (2019) 104138.
- [24] S. Medghalchi, C.F. Kusche, E. Karimi, U. Kerzel, S. Korte-Kerzel, Damage Analysis in Dual-Phase Steel Using Deep Learning: Transfer from Uniaxial to Biaxial Straining Conditions by Image Data Augmentation, *JOM* 72 (12) (2020) 4420–4430.
- [25] C.F. Kusche, F. Pütz, S. Münstermann, T. Al-Samman, S. Korte-Kerzel, On the effect of strain and triaxiality on void evolution in a heterogeneous microstructure—A statistical and single void study of damage in DP800 steel, *Materials Science & Engineering: A* 799 (2021) 140332.
- [26] F. Briffod, T. Shiraiwa, M. Enoki, Micromechanical investigation of the effect of the crystal orientation on the local deformation path and ductile void nucleation in dual-phase steels, *Materials Science & Engineering: A* 826 (2021) 141933.

- [27] J. Liao, J.A. Sousa, A.B. Lopes, X. Xue, F. Barlat, A.B. Pereira, Mechanical, microstructural behaviour and modelling of dual phase steels under complex deformation paths, *Int. J. Plast* 93 (2017) 269–290.
- [28] H.Y. Yu, J.Y. Shen, Design, Evolution of mechanical properties for a dual-phase steel subjected to different loading paths, *Materials* 63 (2014) 412–418.
- [29] S.J. Marcadet, D. Mohr, Effect of compression–tension loading reversal on the strain to fracture of dual phase steel sheets, *Int. J. Plast* 72 (2015) 21–43.
- [30] E.D. Hintsala, U. Hangen, D.D. Stauffer, High-throughput nanoindentation for statistical and spatial property determination, *JOM* 70 (4) (2018) 494–503.
- [31] J. Roa, P.S. Phani, W.C. Oliver, L. Llanes, Mapping of mechanical properties at microstructural length scale in WC-Co cemented carbides: Assessment of hardness and elastic modulus by means of high speed massive nanoindentation and statistical analysis, *Int. J. Refract Metal Hard Mater.* 75 (2018) 211–217.
- [32] M. Sebastiani, R. Moscatelli, F. Ridi, P. Baglioni, F. Carassiti, Design, High-resolution high-speed nanoindentation mapping of cement pastes: Unravelling the effect of microstructure on the mechanical properties of hydrated phases, *Materials* 97 (2016) 372–380.
- [33] C. Tian, D. Ponge, L. Christiansen, C. Kirchlechner, On the mechanical heterogeneity in dual phase steel grades: Activation of slip systems and deformation of martensite in DP800, *Acta Materialia* 183 (2020) 274–284.
- [34] W.C. Oliver, G.M. Pharr, An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, *J. Mater. Res.* 7 (6) (1992) 1564–1583.
- [35] W.C. Oliver, G.M. Pharr, Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology, *J. Mater. Res.* 19 (1) (2004) 3–20.
- [36] T. Blattner, W. Keyrouz, J. Chalfoun, B. Stivalet, M. Brady, S. Zhou, A hybrid CPU-GPU system for stitching large scale optical microscopy images, 2014 43rd International Conference on Parallel Processing, IEEE, 2014, pp. 1–9.
- [37] J. Chalfoun, M. Majurski, T. Blattner, K. Bhadriraju, W. Keyrouz, P. Bajcsy, M. Brady, MIST: accurate and scalable microscopy image stitching tool with stage modeling and error minimization, *Sci. Rep.* 7 (1) (2017) 1–10.
- [38] P. Ludwik, Fließvorgänge bei einfachen Beanspruchungen, in: P. Ludwik (Ed.), *Elemente der Technologischen Mechanik*, Springer Berlin Heidelberg, Berlin, Heidelberg, 1909, pp. 11–35.
- [39] K. Zhang, J. Bai, D. Francois, Numerical analysis of the influence of the Lode parameter on void growth, *Int. J. Solids Struct.* 38 (32–33) (2001) 5847–5856.
- [40] I. Barsoum, J. Faleskog, Micromechanical analysis on the influence of the Lode parameter on void growth and coalescence, *Int. J. Solids Struct.* 48 (6) (2011) 925–938.
- [41] I. Barsoum, J. Faleskog, Rupture mechanisms in combined tension and shear—Experiments, *Int. J. Solids Struct.* 44 (6) (2007) 1768–1786.
- [42] J.R. Rice, D.M. Tracey, On the ductile enlargement of voids in triaxial stress fields, *J. Mech. Phys. Solids* 17 (3) (1969) 201–217.
- [43] Y. Bao, T. Wierzbicki, On the cut-off value of negative triaxiality for fracture, *Eng. Fract. Mech.* 72 (7) (2005) 1049–1069.
- [44] L. Sun, R. Wagoner, Proportional and non-proportional hardening behavior of dual-phase steels, *Int. J. Plast* 45 (2013) 174–187.
- [45] X. Li, C. Liebsch, G. Hirt, J. Lohmar, Modelling of void healing in hot rolling due to recrystallization, *Prod. Eng.* 14 (1) (2020) 43–52.
- [46] C. Park, D.-Y. Yang, A study of void crushing in large forgings I: Bonding mechanism and estimation model for bonding efficiency, *J. Mater. Process. Technol.* 57 (1–2) (1996) 129–140.
- [47] C. Park, D.-Y. Yang, A study of void crushing in large forgings: II, Estimation of bonding efficiency by finite-element analysis, *Journal of Materials Processing Technology* 72 (1) (1997) 32–41.

Chapter 7

Publication 3

On the plasticity of MnS at elevated temperatures and its influence on damage prevalence

M.A. Wollenweber¹, J. Werner², C.F. Kusche¹, C. Tian¹, P.-L. Sun¹, J. Gerlach³, T. Al-Samman¹, S. Korte-Kerzel¹

¹Institute of Physical Metallurgy and Materials Physics, RWTH Aachen University, Germany

²Central Facility for Electron Microscopy, RWTH Aachen University, Germany

³Institute of Metal Forming, RWTH Aachen University, Germany

Acta Materialia 291 (2025) 120987

Received: 29 November 2024

Accepted: 25 March 2025

The original article was distributed under the terms of the Creative Commons CC-BY license.

Maximilian A. Wollenweber: Conceptualisation, Investigation, Data curation, Writing - original draft, Writing - review & editing.

Jonas Werner: Investigation, Writing - review & editing. Carl F. Kusche: Investigation, Writing - review & editing. Chunhua Tian: Investigation Pei-Ling Sun: Investigation, Writing - review & editing. Jannik Gerlach: Investigation, Writing - review & editing. Talal Al-Samman: Supervision, Project administration, Funding acquisition, Writing - review editing. Sandra Korte-Kerzel: Conceptualisation, Supervision, Project administration, Funding acquisition, Writing - review editing.

(Online availability: [j.actamat.2025.120987](https://doi.org/10.1016/j.actamat.2025.120987))



Full length article

On the plasticity of MnS at elevated temperatures and its influence on damage prevalence

Maximilian A. Wollenweber^a,^{*}, Jonas Werner^b, Carl F. Kusche^a, Chunhua Tian^a,
Pei-Ling Sun^a, Jannik Gerlach^c, Talal Al-Samman^a, Sandra Korte-Kerzel^a

^a Institute of Physical Metallurgy and Materials Physics, RWTH Aachen University, 52074 Aachen, Germany

^b Central Facility for Electron Microscopy, RWTH Aachen University, 52074 Aachen, Germany

^c Institute of Metal Forming, RWTH Aachen University, 52074 Aachen, Germany

ARTICLE INFO

Keywords:

Damage initiation

Micromechanics

Scanning Electron Microscopy

Yield phenomena

MnS

ABSTRACT

Forming-induced damage strongly influences the service life and mechanical properties of components made from 16MnCrS5 steel. This damage is initiated in the vicinity of MnS inclusions, which fracture or delaminate at their interfaces due to the mechanical contrast to the steel matrix. Forming processes are often conducted at elevated temperatures; however, the plasticity of MnS at these temperatures in correlation with crystallographic orientation have not been fully explored. In this study, we aim to uncover the high-temperature properties of MnS using micropillar compression and TEM analysis. We then relate them to damage prevalence observed in steel at elevated temperatures with high resolution SEM imaging in combination with AI-assisted damage analysis. We demonstrate that the mechanical contrast of MnS and steel influences the damage prevalence significantly showing a minimum at 400 °C. We additionally determine the CRSS for the primary $\{110\}\langle 1\bar{1}0\rangle$ slip system in the temperature range from 20 °C to 600 °C and the CRSS for the secondary $\{111\}\langle 1\bar{1}0\rangle$ slip system at temperatures of 400 °C and 600 °C. We show that MnS exhibits a yield strength anomaly at 400 °C in specific orientations and at slow loading rates that is accompanied by a change of the active slip system. We relate this yield strength anomaly to thermally activated cross-slip and increased impurity mobility at elevated temperatures.

1. Introduction

Despite centuries of optimisation, modern steel research continues to seek performance improvements. In particular the urgency of anthropogenic climate change has become a new driving force to pursue materials and methods with longer service life times and less environmental impactful pollution. This is a substantial challenge, as metal production, particularly steel manufacturing, plays a sizeable role regarding greenhouse gas emissions [1,2]. Proposed solutions include carbon capture and hydrogen strategies [3,4]; however, their application is unlikely to be widespread in the near future, so there is an additional need to reduce the number of components and material usage in order to lessen the environmental impact. Consequently, the steel industry also makes efforts to focus on minimising emissions and energy consumption by extending service life and promoting lightweight design [5,6].

One effective approach to achieve both of these goals simultaneously, is the control of deformation-induced damage [7–10]. This

study focuses on micrometre-sized voids, which nucleate during forming processes due to contrasting mechanical properties of constituent phases [11–13] and negatively impact mechanical properties, potentially leading to premature component failure [14–16]. Reducing the number and size of these damage sites can enhance the performance of final components [17,18]. This also holds for the case-hardening steel 16MnCrS5 investigated in this work, where most damage sites during forming are caused in the vicinity of manganese sulphide (MnS) inclusions [19,20].

α -MnS, which is the stable polymorph of MnS with the rocksalt crystal structure [21], is known to be able to deform plastically, although it is prone to fracture as inclusions in steels [22,23]. In these cases, its plastic deformation is mostly attributed to dislocation slip on its main slip system $\{110\}\langle 1\bar{1}0\rangle$, although the $\{100\}\langle 1\bar{1}0\rangle$ and $\{111\}\langle 1\bar{1}0\rangle$ slip systems have also been reported as secondary slip systems and synchroshear-induced twins have been reported as an additional mechanism of plastic behaviour at temperatures above 800 °C [24]. The

^{*} Corresponding author.

E-mail address: wollenweber@imm.rwth-aachen.de (M.A. Wollenweber).

<https://doi.org/10.1016/j.actamat.2025.120987>

Received 29 November 2024; Received in revised form 25 March 2025; Accepted 25 March 2025

Available online 4 April 2025

1359-6454/© 2025 The Author(s). Published by Elsevier Inc. on behalf of Acta Materialia Inc. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

Table 1
Chemical composition of 16MnCrS5 case-hardening steel in wt. %.

Material No.	US-Standard	C	Si	Mn	S (max)	Cr
1.7139	AISI 5115	0.14–0.19	0.4	1.0–1.3	0.02–0.04	0.8–1.1

hardness of ionic crystals with the rocksalt crystal structure as a function of temperature and loading rate is known to be influenced by cross-slip and impurity content [25–27]. The micro-mechanical properties in relation to the active slip system of MnS have been studied at ambient temperatures, but their relevance for forming processes at elevated temperatures remains unexplored. This represents a significant knowledge gap, as ambient conditions do not always apply to forming operations, either because the sample is heated up during hot forming [28], or experiences a temperature increase due to friction in processes like extrusion [29]. The mechanical properties of inclusions and the surrounding steel matrix may not necessarily change uniformly with temperature, as is the case with MnS in steel [30], yet they are essential for understanding temperature-dependent damage behaviour and performing accurate damage simulations. Assuming that damage nucleation is largely governed by this mechanical contrast [31,32], we anticipate a shift in damage prevalence with temperature in 16MnCrS5 steel.

Previous work on the mechanical behaviour of MnS as a function of temperature has primarily focused either on polycrystalline MnS bulk samples or single crystals oriented along [100], often conducting tests at large scales unrelated to the size of inclusions present in case-hardening steel [25,33–35]. Furthermore, it remains unclear how the change of plasticity in MnS in turn influences the damage prevalence, as damage was not considered in these works. This study aims to fill that gap by systematically examining the plasticity of MnS in relation to its crystallographic orientation and at a scale relevant for damage initiation in steels. In addition, we will correlate the determined mechanical properties of MnS with those of 16MnCrS5 case-hardening steel to explain the observed temperature-dependent damage prevalence based on variations in critical stresses and mechanical contrast. For this purpose, we start with interrupted tensile tests of 16MnCrS5 steel at elevated temperatures to induce microstructural damage and determine the yield strength. Subsequently, we conduct SEM panoramic imaging and deploy a machine-learning algorithm to assess damage prevalence. This workflow and the trained machine learning algorithm have previously been successfully applied to the same material at ambient temperatures [19]. To determine the active slip systems of MnS, their interactions, and critical resolved shear stresses at elevated temperatures and different rates, we perform micropillar compression experiments and TEM analyses on specially prepared coarse-grained MnS samples. By integrating all obtained results, we can trace changes of mechanical contrast over temperature to explain the observed variations in damage prevalence.

2. Materials and methods

2.1. Case-hardening 16MnCrS5 steel

The analysis of damage prevalence at elevated temperatures, in terms of void number density and void size, was conducted on a case-hardening 16MnCrS5 steel with the chemical composition given in Table 1.

It exhibits a ferritic-pearlitic microstructure in which elongated MnS inclusions are embedded. These MnS inclusions can reach lengths of several tens of micrometres but typically have diameters of up to 2 μm , as detailed in Figs. 1 and 4. All experiments were performed on the as-received state of the material, meaning no additional forming steps were conducted and the steel is not case-hardened.

2.2. Damage quantification

Damage quantification was conducted on dog-bone shaped tensile test samples, machined from the as-received 16MnCrS5 steel billet. The samples were first separated using water jet cutting and then milled to their final geometry. The orientation of MnS stringers in these samples was consistently parallel to the stress direction of the tensile test. Once machined, the samples were mounted in a dilatometer (DIL 805A/D/T, TA instruments, Inc.) equipped with a tensile test module for interrupted tensile testing. A total of four samples were heated to respective temperatures of 20 °C (ambient temperature), 200 °C, 400 °C and 600 °C and then deformed to an approximate length change of 1500 μm to facilitate damage site nucleation in the microstructure. After deformation, the sample was metallographically prepared by grinding with SiC sandpaper and mechanical polishing with water-based diamond suspensions up to 1 μm . In a final step, the sample surface was polished once more with a 0.25 μm oxide polishing solution (OPS) to further improve the surface quality. Once the surface was flat and free of scratches, the sample was mounted in the Scanning electron microscope (SEM) chamber (LEO 1530, Carl Zeiss Microscopy GmbH, Oberkochen, Germany) for secondary electron (SE) panoramic imaging of the sample's centre. During panoramic imaging, multiple images with a lateral resolution of 32 nm/px (horizontal field width of 100 μm and 3072 px) were captured and subsequently stitched together using the Image Composite Editor software (Microsoft Corporation, Redmond, Washington), to create a seamless panoramic image spanning an area of roughly 0.6 mm². The large panoramic image was then analysed with a machine learning algorithm further described in [19], which identifies the MnS inclusions in the microstructure and classifies damage sites related to the inclusions. The complete process, from tensile deformation to damage analysis is described in Fig. 1.

The output of the machine learning assisted damage analysis allows to calculate the Void Area Fraction (VAF) as well as the Mean number of Damage sites per Inclusion (MDI), which are the metrics that will be used to assess damage prevalence, to remain consistent with previous work [19].

2.3. MnS bulk sample

Because the size of the MnS inclusions found in the steel samples is too small to perform systematic micromechanical tests, a bulk MnS sample was fabricated from MnS powder (99.9% purity, ThermoFisher, Kandel, Germany). For this purpose, a crucible made of magnesium oxide (MgO) was filled halfway with MnS powder and heated using an inductive heating source until reaching approximately 1650 °C, close to the melting point of MnS [36]. After a short holding time of 15 min the sample was air cooled, resulting in a bulk MnS sample. This process is also shown in Fig. 2.

After cooling, the sample was adhered to the crucible walls, making separation impossible. The crucible and sample were therefore cut with a diamond saw to create a plane surface, which was prepared similarly to the steel samples, but with longer polishing times. Once the surface was flat and scratch-free the samples were mounted in the SEM (LEO 1530 from Carl Zeiss Microscopy GmbH) chamber for imaging. The larger grains of the bulk MnS sample are shown in the SE micrograph in Fig. 2, enabling multiple micromechanical tests across various orientations and temperatures. In addition to a substantial matrix phase, a second darker phase and several grain boundaries with different contrasts were observed. Energy-dispersive X-ray spectroscopy (EDS) (see Fig. 2) revealed that these correspond to MgO inclusions and an magnesium/oxygen enrichment at the grain boundaries.

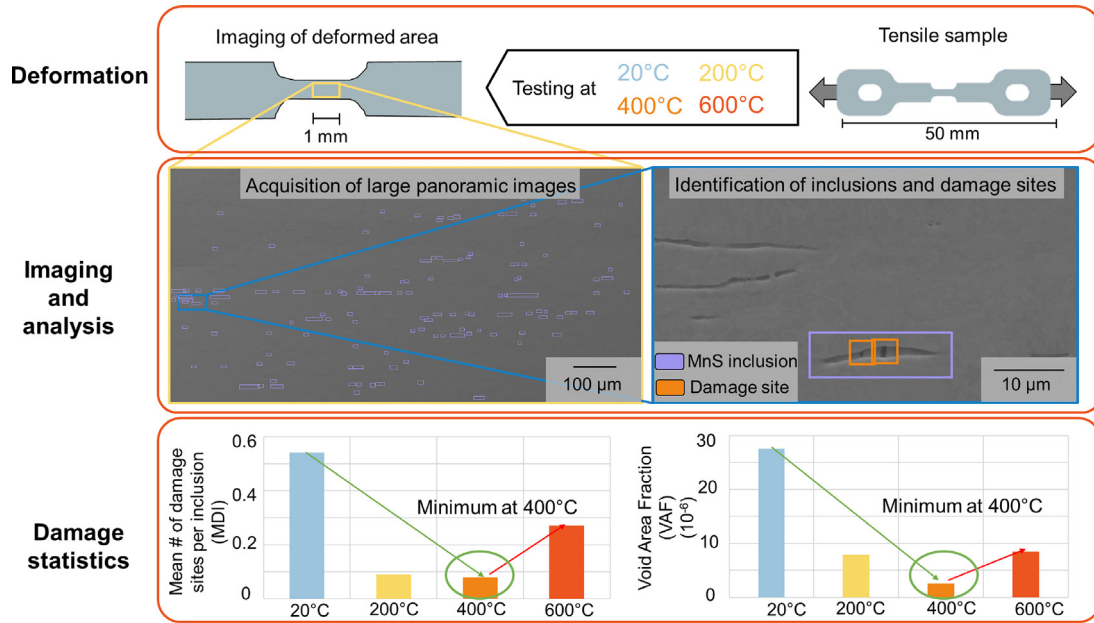


Fig. 1. Workflow and results of damage analysis in 16MnCrS5 case-hardening steel in the temperature range from 20 °C to 600 °C.

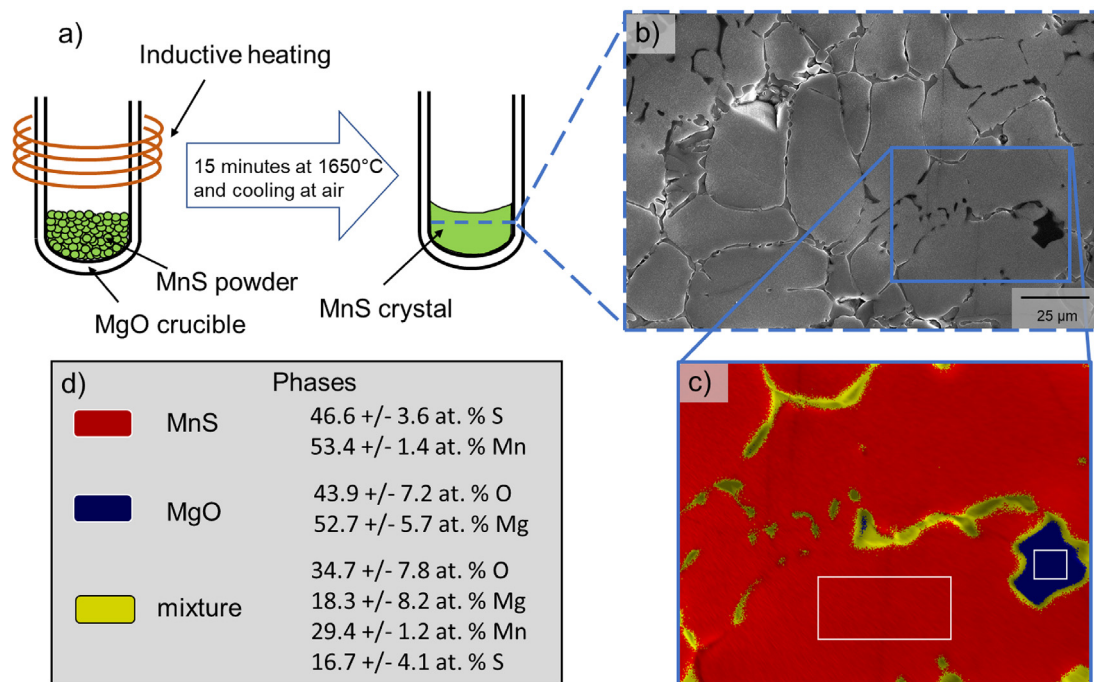


Fig. 2. (a) Fabrication of MnS bulk sample from MnS powder using inductive heating. (b) SE image of the polished surface showing the grain structure and inclusions from the bulk sample. The blue inset corresponds to the area where an EDS measurement was conducted as shown in (c). (d) Chemical composition of the phases detected in the EDS map. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

2.4. Micropillar compression

To determine the active slip systems of MnS at different temperatures and their respective Critical Resolved Shear Stresses (CRSS), we chose three grains with different orientations located near the edges of the standard stereographic triangle. Corresponding EBSD maps represented in terms of inverse pole figure (IPF) colouring are shown in Fig. 3.

Micropillars were fabricated in all three grains using a focused ion beam with a gallium ion source in a Dual-Beam FIB (Helios NanoLab 600i from FEI Inc.). Rough milling was carried out first with an initial

beam current of 9.3 nA, followed by successive milling steps using lower beam currents before proceeding into the final step at a low current of 80 pA. The pillars were designed to have dimensions of 4 µm in height and 1.8 µm in diameter; however, the actual measurements were used for subsequent calculations. To ascertain these dimensions and to ensure that each micropillar was single-crystalline and free of grain boundaries or defects, we imaged them from the top and four side angles using SEM prior to compression testing. Grain boundaries in the sample exhibited strong contrast under SEM, enabling their detection. Only micropillars without visible grain boundaries or defects were selected for mechanical measurements. Pillars were found to have a

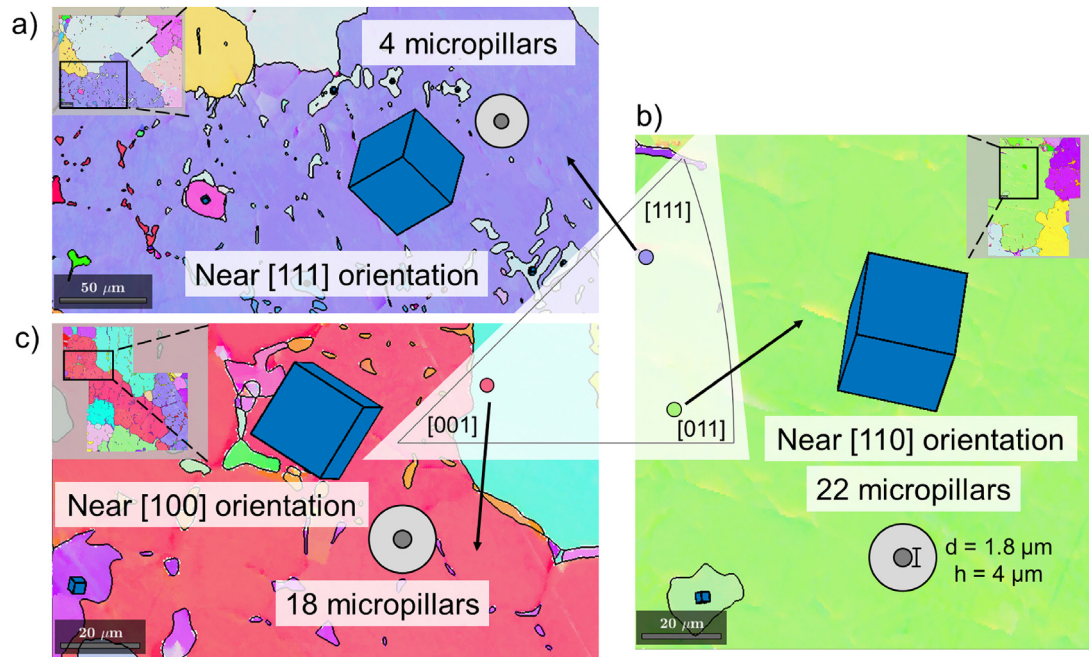


Fig. 3. Selected areas of the EBSD-IPF maps for the three grains in the bulk MnS sample chosen for micropillar compression: near $[111]$ orientation in (a), near $[110]$ orientation in (b), and near $[100]$ orientation in (c). Complete maps for all grains are given in the top left corner insets. Pillar dimensions relative to the grain size are presented as grey circles in the EBSD maps. The grain orientations relative to the pillar geometry are visualised by cubic unit cells. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

taper in the range of 2° to 4° , which was shown to have a negligible effect on the plastic behaviour [37]. Once all pillars were imaged the sample was mounted in an in-situ nanoindentation device (inSEM from Nanomechanics Inc.) equipped with a high temperature load head which was then placed within the chamber of a SEM (Clara from TESCAN) to prepare for the compression testing. The sample and indenter tip were heated successively from 20°C to 200°C , 400°C and 600°C to align with the temperatures used for damage analysis (Section 2.2). At each temperature setting, pillars from all orientations were compressed at a constant loading rate of 0.02 mN s^{-1} before the temperature was increased again, while ensuring that each individual pillar was only loaded at one temperature. In the orientation near $[110]$ a second set of pillars was tested at a higher loading rate of 0.2 mN s^{-1} at temperatures from 20° to 400° and a third set of pillars was tested at an even higher loading rate of 1 mN s^{-1} at temperatures of 20°C and 400°C . Prior to testing, it was ensured that both the tip and sample temperatures matched closely to minimise any thermal expansion effects on the force-displacement curve. In total, 64 micropillars were milled and compressed throughout this work.

After compression, the pillars were imaged once more from the same angles as prior to deformation. Here, the slip traces on the surface of the pillar were of particular interest, as they provide insight into the active slip systems. These traces were compared to slip steps on simulated pillars to determine the active slip system. The workflow of this procedure has been shown before by Heller and Gibson [38,39] and is expanded upon in more detail in the Supplementary Materials.

After determining the active slip system, the force-displacement data from the micropillar compression tests was converted into engineering stress and strain data using the height and top diameter measurements taken before deformation. The data was evaluated at 1% and 5% plastic strain. The exact workflow of determining the stress is further expanded upon in the Supplementary Materials. The Schmid factor and the determined yield stress of the micropillar at 1% plastic strain were then utilised to calculate the CRSS for each individual slip system using Schmid's law. The analysis of active slip systems along with the calculation of the CRSS was conducted for every pillar exhibiting a clear slip step.

2.5. TEM analysis

Even though most micropillars showed a clear slip step that could be assigned to one slip system, some pillars showed a more ambiguous behaviour. To confirm the active slip plane for these cases, Transmission Electron Microscopy (TEM) analyses were conducted on one pillar with a near $[110]$ orientation. The TEM lamella was extracted such that the suspected slip planes $(0\bar{1}1)$ and $(11\bar{1})$ could be viewed edge-on by tilting of the lamella within the TEM. The lamella was prepared using a ZEISS Crossbeam 350 FIB/SEM (Carl Zeiss Microscopy GmbH) and analysed with (scanning) transmission electron microscopes ((S)TEM) Tecnai F20 (FEI Company) equipped with a STEM bright field/annular dark field (BF/ADF) detector and a Veleta S04F camera (EMSIS GmbH) at 200 kV. Furthermore, high resolution TEM (HRTEM) analysis of the MnS micropillar in a $\langle 110 \rangle$ zone axis was performed at 200 kV with a JEOL JEM-F200 TEM (Jeol Ltd., Japan) equipped with a Gatan OneView (Gatan Inc., Pleasanton, CA, USA) CMOS camera.

3. Results

3.1. Tensile tests at ambient and elevated temperatures

The main outcome of the characterisation work carried out on the tensile samples strained at elevated temperatures is the damage quantification via the parameters AVF and MDI for each test temperature, as shown in the lower part of Fig. 1. Both damage parameters show a similar trend, with their maximum value at 20°C followed by a decrease until 400°C . At 400°C both parameters attain a minimum before increasing again from 400°C to 600°C . In addition to these quantitative measures, detailed images of individual damage sites were also captured to evaluate the damage nucleation mechanisms. In Fig. 4 a selection of examples depicting damage sites and MnS inclusions for each temperature are presented.

Although the extent of damage varies with temperature, the size and shape of MnS inclusions remain unchanged, consistently exhibiting their characteristic stringer form. Furthermore, the observed damage mechanisms remain unchanged across temperatures of 20°C , 200°C

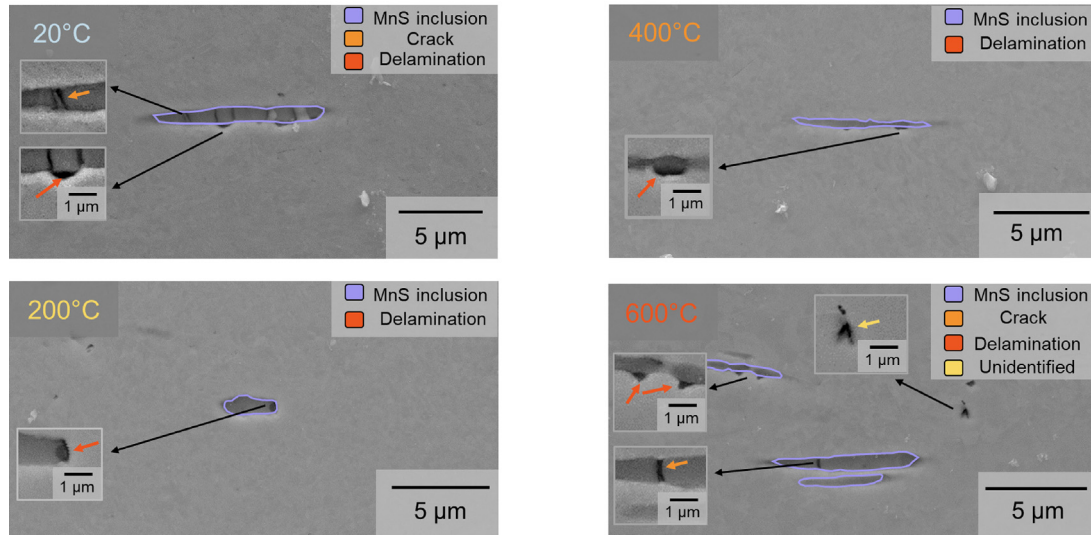


Fig. 4. Selected areas of damage sites from the panoramic images taken at 20 °C, 200 °C, 400 °C and 600 °C with MnS inclusions outlined in purple. Insets show different features of the damage types associated with these inclusions. The colour of the arrows indicates the initiation mechanisms: Orange for MnS cracking, red for interface delamination, and yellow for damage sites not linked to MnS inclusions. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 2

Flow stress at 1% strain of 16MnCrS5 case-hardening steel at different temperatures.

Temperature	20 °C	200 °C	400 °C	600 °C
1% flow stress (MPa)	430	357	347	214

and 400 °C. However, at 600 °C an exception arises, as voids additionally initiate outside the vicinity of MnS inclusions (highlighted in yellow in Fig. 4), revealing an additional unidentified damage initiation mechanism. Damage sites belonging to this mechanism are significantly fewer in number compared to those related to MnS inclusions and are not considered for the calculation of AVF or MDI parameters in this work. In addition to the microstructural data, the mechanical data from the tensile tests were evaluated at a flow stress of 1% strain for each temperature. These flow stresses are summarised in Table 2 and indicate a non-linear decrease with rising test temperature.

3.2. Micropillar compression experiments

The engineering stress–strain curves, distinguished by orientation, temperature, and loading rate and with highlighted stresses at 1% and 5% plastic strain are shown in Fig. 5 up to a strain of 25%. It is apparent that the stress–strain curves differ substantially with both orientation, temperature and loading rate. At comparable loading rates maximum stresses are observed for the near [111] orientation, while the near [100] orientation was found to generally exhibit the lowest stress levels. The flow stress was also observed to decrease with increasing temperature across all orientations, except for micropillars oriented near [110] and [111] compressed at 400 °C, where an increase in stress is noted compared to 200 °C at low loading rates. For the near [100] orientation, no clear trend regarding the effect of temperature on the flow stress can be discerned from the stress–strain curves alone.

Complementary to the mechanical data, the active slip systems for each micropillar were determined and are summarised in Table 3. In total, two different slip systems were observed to be activated across all temperatures and orientations; i.e. $\{110\}\langle 1\bar{1}0\rangle$ and $\{111\}\langle 1\bar{1}0\rangle$. At lower temperatures (20 °C to 200 °C), the $\{110\}$ slip system is active in all three orientations at all loading rates. Starting from 400 °C, a transition to the $\{111\}$ slip plane is observed for the deformed micropillars oriented near [110] (Fig. 6a). This change of slip system is only observed at the lowest loading rate of 0.02 mN s^{−1} and

Table 3

Summary of activated slip planes for each orientation, temperature and loading rate. The slip directions remains consistent for all orientations and directions as $\langle 1\bar{1}0\rangle$.

Orientation	20 °C	200 °C	400 °C	600 °C	Loading rate
Near [100]	$\{110\}$	$\{110\}$	$\{110\}$	$\{110\}$	0.02 mN s ^{−1}
Near [110]	$\{110\}$	$\{110\}$	$\{111\}$	$\{111\}$	0.02 mN s ^{−1}
Near [110]	$\{110\}$	$\{110\}$	$\{110\}$	–	0.2 mN s ^{−1}
Near [110]	$\{110\}$	–	$\{110\}$	–	1 mN s ^{−1}
Near [111]	$\{110\}$	$\{110\}$	$\{111\}$	Uncertain	0.02 mN s ^{−1}

at higher loading rates the $\{110\}$ slip system remains active even at 400 °C. For micropillars with a near [100] orientation, the $\{110\}$ slip system persists through the entire temperature range even at the lowest loading rate. However, at high temperatures of 600 °C, determining the active slip system becomes more challenging, as clear slip steps can no longer be observed. Many pillars that exhibit the $\{110\}$ slip system also show signs of double slip on a second $\{110\}$ slip system with an identical Schmid factor as shown in Fig. 6(b). Additionally, for this orientation, another change of slip system is observed from 400 °C to 600 °C. At this temperature, however, the slip system does not change to the $\{111\}$ system; instead, it shifts to a different $\{110\}$ slip system with a lower Schmid factor (Fig. 6(c)).

By multiplying the Schmid factor acquired from determining the active slip systems with the corresponding stress at 1% plastic strain, the CRSS values were calculated for each orientation (cf. Tables Table 6, Tables 5 and 4). Table 7 combines these values for all orientations at a loading rate of 0.02 mN s^{−1}, distinguishing them only by active slip system.

Near [100] orientation: For this orientation, the $\{110\}$ slip system is observed at all temperatures, exhibiting a trend of decreasing CRSS with increasing temperature (Table 4). The only exception occurs at 400 °C, where the CRSS appears to increase.

Near [110] orientation: For this case at a loading rate of 0.02 mN s^{−1}, the $\{110\}$ slip system is activated at both 20 °C and 200 °C with obvious decrease in the CRSS at 200 °C (Table 5). At 400 °C and 600 °C, however, the $\{111\}$ slip system becomes active, demonstrating a higher CRSS. At 400 °C, the respective CRSS is almost twice as high as that of the $\{110\}$ slip system at 200 °C. Similar to the behaviour of the $\{110\}$ slip system, the CRSS of the $\{111\}$ slip system also decreases with increasing temperature from 400 °C to 600 °C. At faster loading rates the $\{110\}$ slip system remains as the active slip system throughout the

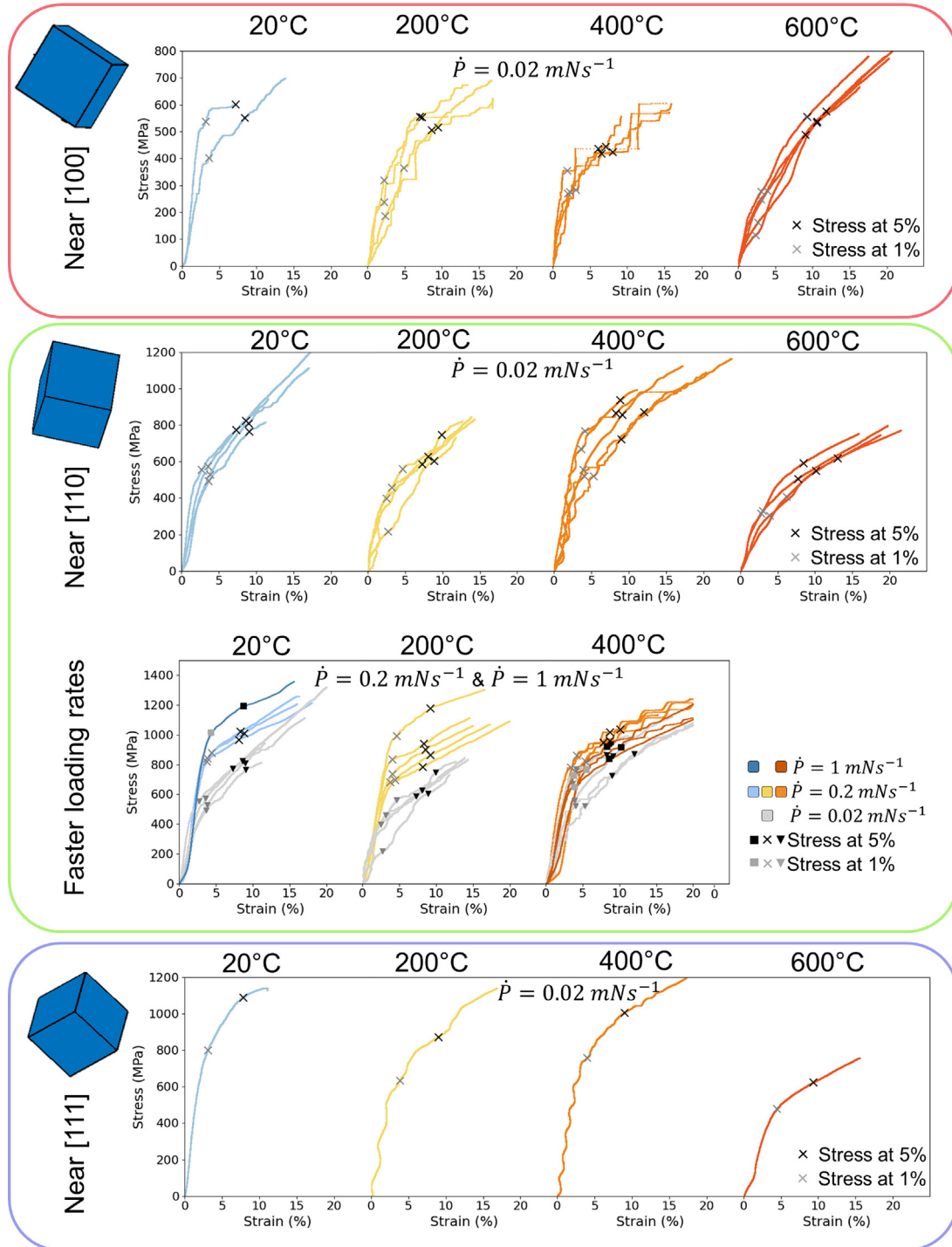


Fig. 5. Engineering stress-strain curves of micropillar compression experiments up to a strain of % with highlighted stresses at 1% and 5% plastic strain. The curves are differentiated by temperature, orientation and loading rate. In the orientation near [110] at faster loading rates, the curves from slow loading rates of the same orientation are also given in grey. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

complete temperature range investigated and its CRSS increases for all temperatures except for 400 °C. Here, the CRSS lies in the same region for all loading rates.

Near [111] orientation: This orientation exhibits a trend similar to the low loading rate in the previous orientation, with the {110} slip system activated at both ambient temperature and 200 °C (Table 6). At 400 °C, the {111} slip system is activated at a much higher CRSS compared to the {110} slip system. However, due to difficulties in conducting a reliable slip system analysis at 600 °C, no CRSS is calculated for this temperature. Comparing the CRSS values at 20 °C and 200 °C with those

of the [100] and [110] orientations reveals that they are relatively close to each other. An overview of the CRSS values, independent of pillar orientation, is presented in Table 7 for both {110} and {111} slip systems as a function of temperature.

3.3. Confirmation of slip plane with TEM

In select cases, where it was difficult to assign a slip trace to either a {111} or {110} slip plane due to high deformation and inconclusive comparisons with simulations, in-depth TEM analysis was employed.

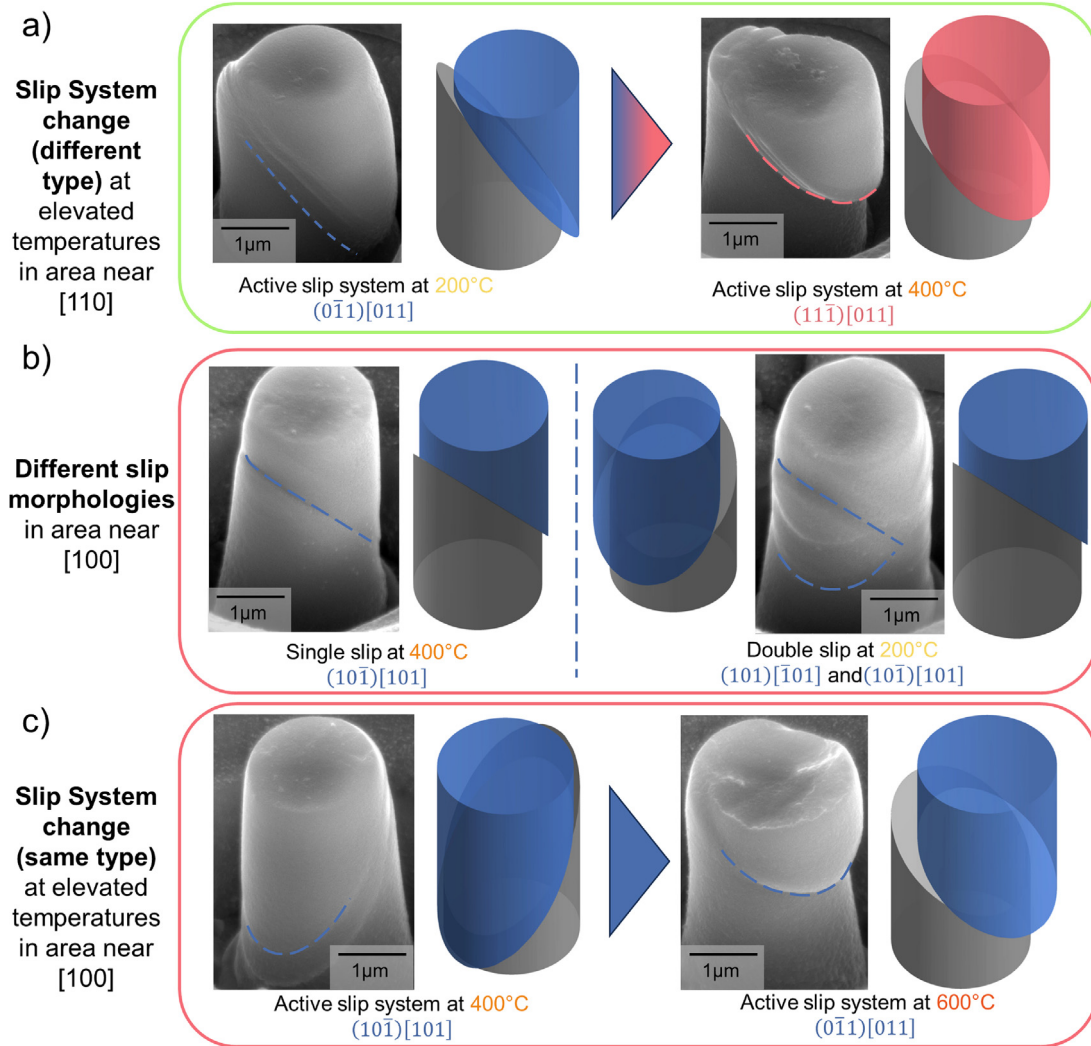


Fig. 6. Compressed micropillars with their respective slip system activation. (a) Transition of active slip system from 200 °C to 400 °C for the near [110] orientation. (b) Two pillars with a near [100] orientation exhibit double slip and single slip. (c) Transition of active slip system from 400 °C to 600 °C. The angle of view changes from (a) through (c) in accordance with the slip trace orientation but remains consistent in each sub-figure.

Table 4

CRSS of observed slip systems at different temperatures for the near [100] orientation at a loading rate of 0.02 mN s^{-1} .

Temperature	Slip system	CRSS (MPa)
20 °C	$\{110\} \langle 1\bar{1}0 \rangle$	219 ± 44
200 °C	$\{110\} \langle 1\bar{1}0 \rangle$	144 ± 26
400 °C	$\{110\} \langle 1\bar{1}0 \rangle$	142 ± 21
600 °C	$\{110\} \langle 1\bar{1}0 \rangle$	98 ± 33

An example is provided in Fig. 7a), illustrating a micropillar (near [110] orientation) deformed at 400 °C, along with the potential slip systems. The orientation of the TEM-lamella was chosen such that it allows both potential slip planes to be viewed edge on. The main slip plane was confirmed to be the $(11\bar{1})$ plane when it was viewed edge-on and sharp differences in contrast could be distinguished from diffuse bending contours (Fig. 7b). In two beam condition of the 222 reflex reached from the $[2\bar{1}\bar{1}]$ zone axis, dislocations appeared as individual lines within the $(0\bar{1}1)$ plane (Fig. 7c). These results provide clear evidence of plastic deformation occurring on $\{111\}$ planes, likely transitioning from the $\{110\}$ slip plane at 400 °C (cf. Table 5). Fig. 7(d) shows HRTEM images obtained at zone axis $[\bar{1}0\bar{1}]$ in which the $(\bar{1}11)$, $(11\bar{1})$ and (010) planes are edge-on. The lattice image shows the presence of some nano-twins with the twin plane on the $(\bar{1}11)$ plane. The fast

Table 5

CRSS of observed slip systems at different temperatures for the near [110] orientation.

Loading rate	Temperature	Slip system	CRSS (MPa)
0.02 mN s^{-1}	20 °C	$\{110\} \langle 1\bar{1}0 \rangle$	172 ± 11
0.02 mN s^{-1}	200 °C	$\{110\} \langle 1\bar{1}0 \rangle$	121 ± 36
0.02 mN s^{-1}	400 °C	$\{111\} \langle 1\bar{1}0 \rangle$	283 ± 43
0.02 mN s^{-1}	600 °C	$\{111\} \langle 1\bar{1}0 \rangle$	161 ± 27
0.2 mN s^{-1}	20 °C	$\{110\} \langle 1\bar{1}0 \rangle$	272 ± 42
0.2 mN s^{-1}	200 °C	$\{110\} \langle 1\bar{1}0 \rangle$	252 ± 41
0.2 mN s^{-1}	400 °C	$\{110\} \langle 1\bar{1}0 \rangle$	256 ± 15
1 mN s^{-1}	20 °C	$\{110\} \langle 1\bar{1}0 \rangle$	324
1 mN s^{-1}	400 °C	$\{110\} \langle 1\bar{1}0 \rangle$	228 ± 19

Fourier transformation (FFT) of the selected square area (marked by the red square) is inset. In the FFT twin diffraction spots are highlighted by the blue rectangle, while the matrix diffraction spots are shown by the red rectangle. Additionally, some diffraction streaks are observed along the $(\bar{1}11)$ plane in the FFT (indicated by the purple arrows) and along the $(11\bar{1})$ plane (not shown here), which indicate the presence of stacking faults on both the $(\bar{1}11)$ and $(11\bar{1})$ planes.

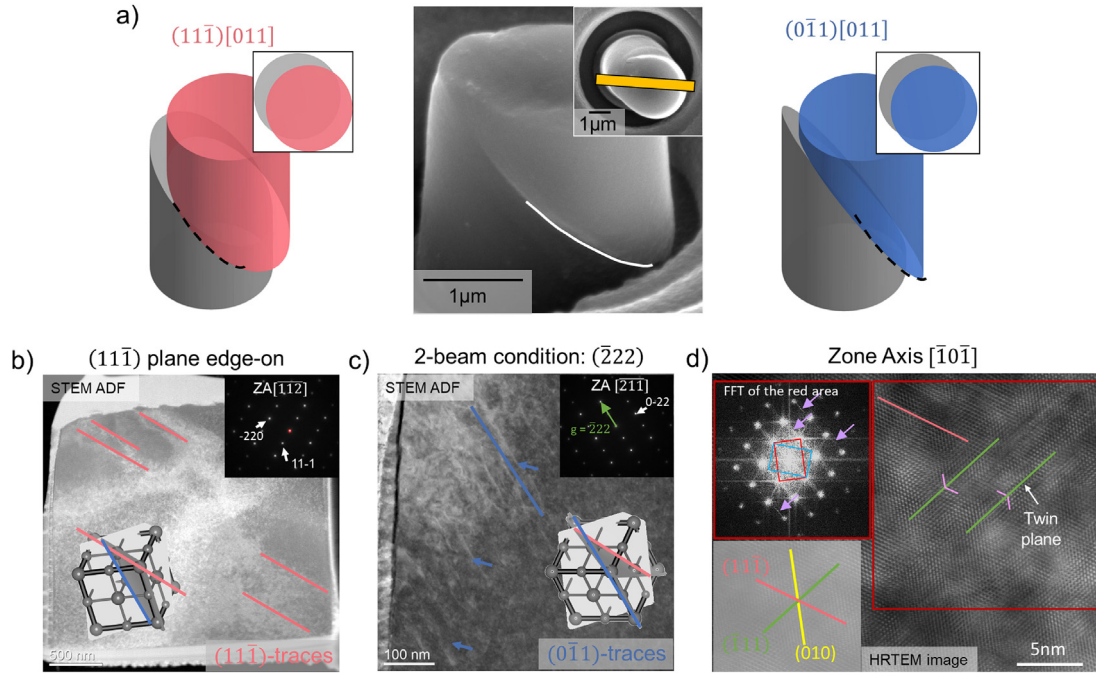


Fig. 7. (a) Deformed micropillar with a highlighted slip plane trace and simulation of potential slip systems associated with that slip trace. (b) STEM ADF (annular dark field) image taken at zone axis $[1\bar{1}\bar{2}]$ showing high concentration of defects on the $(11\bar{1})$ plane, which is edge-on at this zone axis. The SAD pattern of zone axis $[1\bar{1}\bar{2}]$ is given in the inset. A unit cell of MnS created in Vesta [40] along with the $(11\bar{1})$ plane, the $(01\bar{1})$ plane (both in darker grey) and the plane of the lamella (foil plane, in light grey) is also provided. The intersections of the crystallographic planes with the foil plane are highlighted in pink for the $(11\bar{1})$ plane and in blue for the $(01\bar{1})$ plane. (c) STEM ADF image taken under $g = \bar{2}22$ two-beam condition which could be reached from zone axis $[2\bar{1}\bar{1}]$ showing many line features of defects in the $(01\bar{1})$ plane, which is edge-on in zone axis $[2\bar{1}\bar{1}]$. The SAD pattern of zone axis $[2\bar{1}\bar{1}]$ is given in the inset. Here, a second unit cell of MnS with highlighted intersections is given, this time oriented along the $g = \bar{2}22$ two-beam condition. (d) High resolution TEM image taken at zone axis $[1\bar{0}\bar{1}]$ and image of the corresponding fast Fourier transformation (FFT) of the area highlighted in red are shown. A few nano-twins with the twin plane on the $(1\bar{1}\bar{1})$ plane are observed, as highlighted by the diffraction spots marked by the blue rectangle in the FFT image. The red rectangle on the FFT image indicates the diffraction spots for the matrix. The plane traces of $(11\bar{1})$, $(1\bar{1}\bar{1})$ and (010) are also provided. Additionally, some diffraction streaks (marked by the purple arrows) on the FFT image are also noticed, which indicate the presence of stacking faults on the $(1\bar{1}\bar{1})$ plane. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 6

CRSS of observed slip systems at different temperatures for the near $[111]$ at a loading rate of 0.02 mN s^{-1} .

Temperature	Slip System	CRSS (MPa)
20 °C	$\{110\} \langle 1\bar{1}0 \rangle$	165
200 °C	$\{110\} \langle 1\bar{1}0 \rangle$	128
400 °C	$\{111\} \langle 1\bar{1}0 \rangle$	313
600 °C	Not determined	–

Table 7

Overview of the CRSS for different slip systems at different temperatures averaged from all orientations at a loading rate of 0.02 mN s^{-1} .

Slip system	$\{110\} \langle 1\bar{1}0 \rangle$	$\{111\} \langle 1\bar{1}0 \rangle$
Temperature	CRSS (MPa)	CRSS (MPa)
20 °C	165 ± 22	–
200 °C	132 ± 30	–
400 °C	142 ± 21	286 ± 42
600 °C	98 ± 33	162 ± 27

4. Discussion

4.1. Plasticity of MnS

In the majority of deformed pillars, we observed slip on $\{110\}$ planes, with some pillars also exhibiting slip on $\{111\}$ under specific conditions. The activation of the $\{110\}$ system as the main slip system is consistent with earlier studies on the deformation and fracture of MnS single crystals. Secondary slip has been reported on $\{111\}$ planes [22,23], but requires significantly larger stresses because the ionic structure of MnS leads to slip plane dependent variations in

Table 8

Schmid factors of all discussed slip systems, including the ratio of Schmid factors of the primary $\{111\}$ to the primary $\{110\}$ slip system.

Orientation	$\{110\} \langle 1\bar{1}0 \rangle$		$\{111\} \langle 1\bar{1}0 \rangle$	
	Primary	Secondary	Primary	Ratio of primary $\{111\}$ to primary $\{110\}$
Near $[100]$	0.465	0.452	0.470	1.01
Near $[110]$	0.319	0.173	0.472	1.48
Near $[111]$	0.202	0.141	0.413	2.04

the electrostatic effects associated with the ion configuration in the cores of edge dislocations [41]. None of the indexed slip events on the micropillar surfaces were indicative of slip on $\{100\}$. For both primary and secondary slip, $\langle 1\bar{1}0 \rangle$ was identified as the main slip direction, which is expected to involve a low energy barrier resulting from electrostatic interactions between Mn and S ions analogous to NaCl and AgCl [42]. In order to assess the relative critical shear stresses on any hard and soft slip systems, a compilation of all Schmid factors for each slip system and for all three orientations can be found in Table 8. Even though the Schmid factor for slip on the primary $\{111\}$ plane is higher than the Schmid factor for slip on the primary $\{110\}$ plane in all orientations, slip at room temperature was exclusively observed on $\{110\}$ planes. This marks the $\{110\} \langle 1\bar{1}0 \rangle$ slip system as the soft slip system and $\{111\} \langle 1\bar{1}0 \rangle$ as the hard slip system, with a CRSS at least twice that of the soft system based on our measurements with respect to both systems at 400 °C and a loading rate of 0.02 mN s^{-1} (Table 7) and as a lower bound at room temperature based on the Schmid factor ratio.

The consistent identification of slip on $\{110\}$ planes as the primary mechanism for plastic deformation across all deformation conditions, was marked by one exception. This exception occurred in orientations

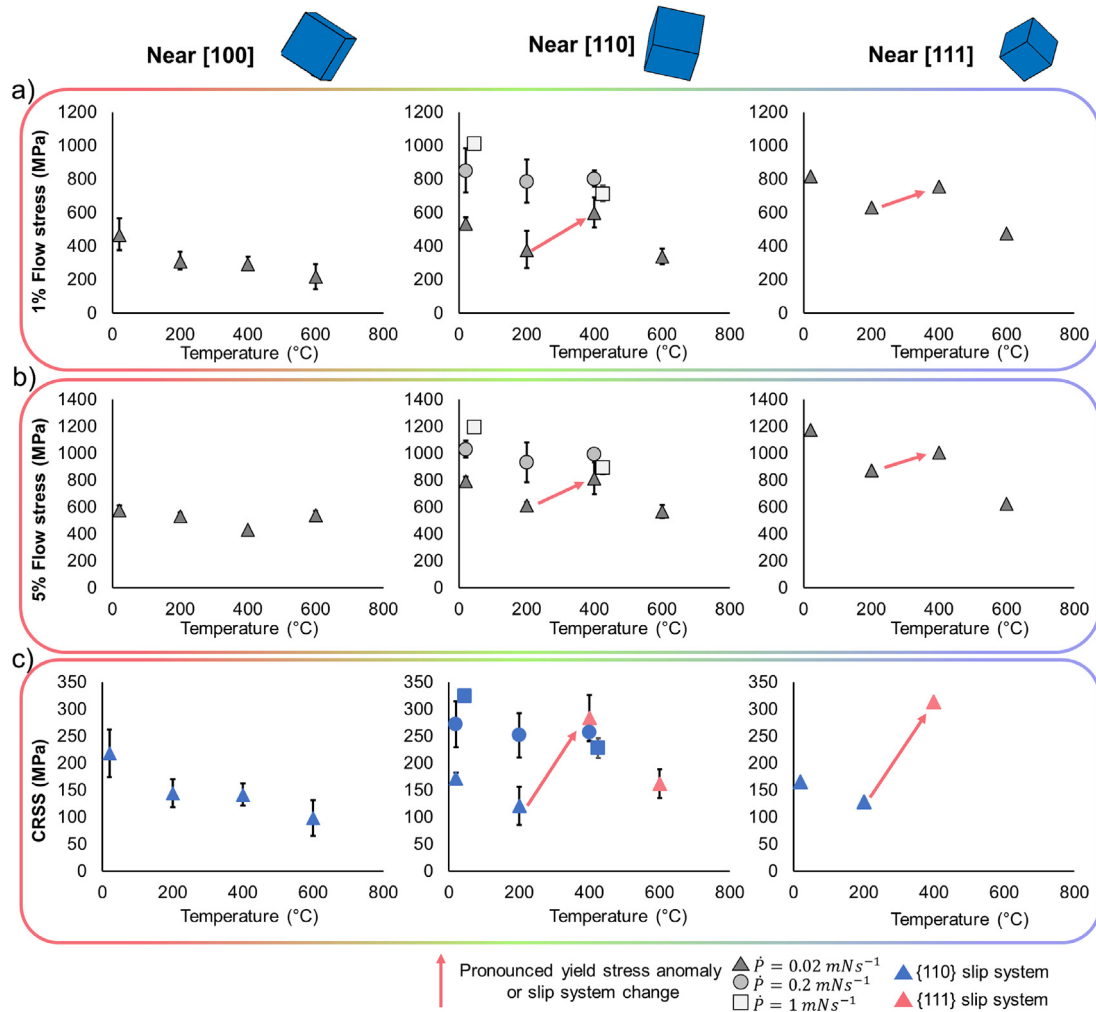


Fig. 8. Trend of flow stress at 1% plastic strain in (a), flow stress at 5% plastic strain in (b) and CRSS in (c) over temperature per orientation and loading rate (cf. Tables 3 and 6 to Table 4). The data points in (c) are coloured according to the observed active slip system. Blue corresponds to the $\{110\}$ and pink to the $\{111\}$ slip system. Pronounced yield stress anomalies and slip system changes have been highlighted using a pink arrow. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

near $[110]$ and $[111]$ under the lowest loading rate and at a temperature of 400 °C. In the subsequent section, we will set aside these specific conditions and first focus exclusively on the remaining possible combinations.

In terms of a quantitative comparison of the stresses measured here and elsewhere, at room temperature, the CRSS is slightly higher than previously reported [22], yet still falls within the expected range for micropillars of similar size and crystal structure [43]. Possible reasons for the differing values could include different solute impurity levels, a slightly lower diameter of the micropillars in this study, as well as the evaluation conducted at 1% plastic strain rather than 0.2% plastic strain. With increasing temperature, the general expectation for ionic crystals like MnS is that resistance to plastic deformation decreases due to thermally activated dislocation motion against the lattice resistance, as observed in isostructural phases [44,45]. This trend is evident in the engineering stress–strain curves presented in Fig. 5 as well as in the stresses at 1% and 5% plastic strain and the CRSS as given in Fig. 8, where an overall decrease in flow stress with rising temperature is observed for most orientations (also cf. Table 7).

For the same mechanism of thermally activated dislocation glide, higher loading rates lead to an increased yield stress at constant mobile dislocation density owing to the increase in imposed dislocation velocity and hence reduced effect of thermally activated kink formation. This is in fact observed here, as evident across the room temperature flow

stresses at 1% and 5% plastic strain measured at different loading rates in the near $[100]$ orientation (Fig. 8 and Table 5). However, the decrease of the CRSS over temperature is not linear, as would be expected for a constant governing deformation mechanism. Instead, the CRSS at 400 °C remains approximately on the same level compared to the CRSS at 200 °C in $[100]$ orientation. Similarly, a diminished dependence of stress on strain rate is observed in the orientation near $[110]$ at 400 °C. At the same time, serrations occur in the stress–strain data for both cases. Their presence is commonly associated with jog formation during cross-slip and/or pinning of dislocations by solute atoms. Both have been observed frequently in MnS and other isostructural crystals. In case of cross-slip, $\{111\}$ planes have been identified as the first cross-slip planes in isostructural NaCl [27] and in $\text{MgO-Al}_2\text{O}_3$ spinel similar mechanical behaviour has also been associated with cross-slip [46]. In particular when cross-slip occurs in the presence of solutes, jog pinning is thought to give rise to hardening and serrated flow [47]. Hardening due to solutes, in particular Ca, has been reported in the temperature range considered here, with a much lower loss in hardness between room temperature and 800 °C than in pure single crystals [33]. When CaO and CaS are present an even stronger hardening effect is observed leading to a near constant hardness from 200 °C onwards up to approximately 600 °C [25]. Addition of Fe to replace Mn on the other hand was found not to have a significant effect on the hardness of MnS.

At 600 °C, slip on $\{110\}$ planes is easy enough for the small difference in Schmid factors between the primary and secondary slip system

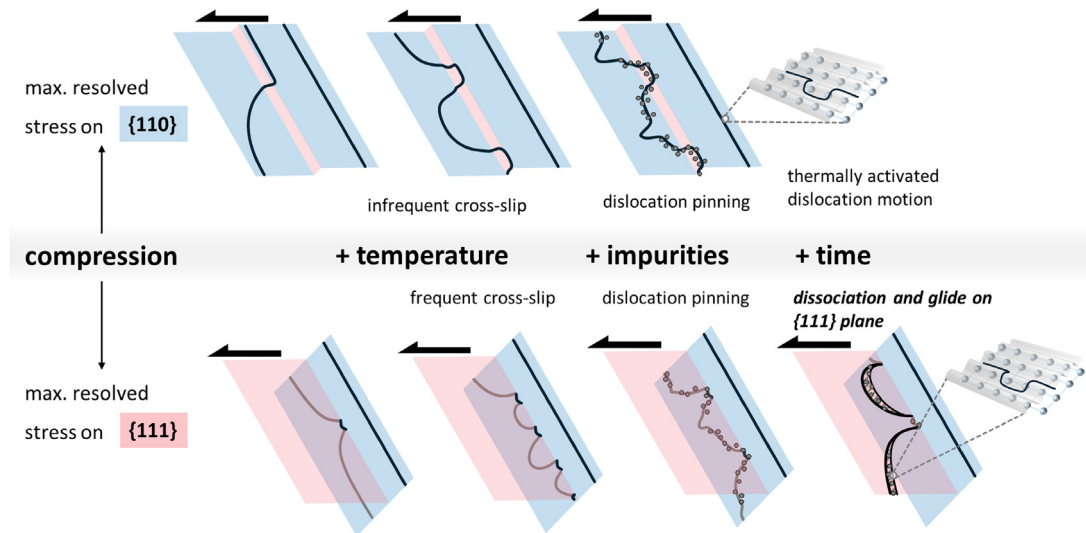


Fig. 9. Proposed scenarios involving impurity mobility and dislocation dissociation leading to experimental observations depending on orientation, temperature, the presence of solutes, and strain rate/time. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

to become negligible near the $[100]$ direction, allowing dislocation glide on different $\{110\}$ planes (Fig. 6c) and with diminishing solute interaction, as evident from the absence of serrations in the compression curves. This activation of multiple intersecting slip planes is likely the cause of the higher strain-hardening rate observed in Fig. 5 and the elevated flow stress at 5% plastic strain shown in Fig. 8 for compression at 600°C in $[100]$ direction.

Our observations are therefore consistent with expectations assuming lattice resistance controlled flow at room temperature and an increasing mobility of impurity atoms with temperature. In this case, the loss of rate sensitivity is equally consistent with other observations of dynamic strain ageing and rate dependence, which is known to approach zero or even negative values also in other materials [48]. The behaviour of dislocations and their interactions with impurity atoms when dislocation glide on $\{110\}$ planes is favoured, is visualised in the top part of Fig. 9. The dislocation is illustrated moving mainly on a $\{110\}$ plane (shown in blue) even though infrequent cross-slip to $\{111\}$ planes (shown in light pink) might occur at elevated temperatures and impurities additionally pin dislocations at elevated temperatures when present. The effect of time, imposed by the strain rate, affects flow predominately in terms of double kink formation and propagation to overcome the Peierls barrier (Fig. 9).

4.2. Yield stress anomaly at elevated temperatures and low loading rates

In contrast to the conditions discussed above, a more pronounced yield strength anomaly is observed at 400°C in micropillars oriented near $[110]$ and $[111]$ and deformed at the lowest loading rate of 0.02 mN s^{-1} . Here, we observed an increase in stress at 400°C compared to 200°C , as illustrated by the pink arrow in Fig. 8. This anomaly is accompanied by a change in the active slip system from the primary $\{110\}\langle 1\bar{1}0\rangle$ to the secondary $\{111\}\langle 1\bar{1}0\rangle$ system. This transition of the active slip plane to $\{111\}$ at 400°C is specific to a combination of three conditions:

- Compression in an orientation, where the Schmid factor for the $\{111\}$ (cross-slip) system is significantly higher than for the primary $\{110\}$ system
- Elevated temperatures
- Slow loading rates

Even though only one observation could be made in the orientation near $[111]$, the slip system change was observed in several pillars in the orientation near $[110]$.

In principle, thermal activation of dislocation motion, solute effects and cross-slip have the same effect on slip on $\{111\}$ planes as on slip on $\{110\}$ planes. However, the specific combination of experimental conditions leads to a synergetic effect, amplifying the increase of resistance to plastic deformation at 400°C . Here, we propose a process that captures the increased resistance to plastic deformation on $\{111\}$ compared to $\{110\}$ as well as the transition of the active slip system as a combination of thermally activated cross-slip and impurity mobility. On their own, these mechanisms are already known to affect the mechanical properties of MnS as a function of temperature [25,33,46], as discussed in the previous section. However, to the best of our knowledge, their combination and effect on mechanical properties has not been previously reported in MnS, likely because of the specific experimental conditions required. These conditions and their effects on a dislocation line are illustrated in the lower half of Fig. 9. Here, the elevated temperature of 400°C , inducing frequent cross-slip in orientations where the maximum resolved shear stress acts on $\{111\}$ planes, and high impurity mobility, as well as low dislocation velocities contribute to an orientation-dependent locking of the $\{110\}$ slip system. This leads to a change in the active slip system, in spite of the higher CRSS of the new slip system, and an increased resistance to plastic deformation. We focus here on elucidating the interplay of dislocation dissociation and increased impurity mobility that lead to the locking of the slip plane in Fig. 10.

4.2.1. Orientation + time - cross-slip and dissociation:

Due to the lower CRSS, dislocations should initially be more mobile on the $\{110\}$ plane. However, with negligible or small dissociation width, elevated temperatures result in frequent cross-slip to the more highly stressed $\{111\}$ plane (top left of Fig. 10), where, dislocations can dissociate (top right of Fig. 10). This has been observed in MnS at temperatures between 800°C and 1200°C [24] and also indirectly by nanotwin formation in our TEM analysis Fig. 7(d). As a consequence of frequent cross-slip and dissociation on the cross-slip plane, dislocations cannot reverse cross-slip and are also unable to move on the cross-slip $\{111\}$ plane, as the CRSS for this plane has not yet been reached.

4.2.2. Temperature + time - increased impurity mobility:

At 400°C , impurities gain increased mobility and the prolonged waiting times of dislocations after their initial cross-slip, due to a low loading rate, allow impurities to accumulate in the stacking fault [49], presumably increasing dissociation width, or enabling dissociation onto adjacent planes into synchro-shear partials [24] (bottom right of Fig.

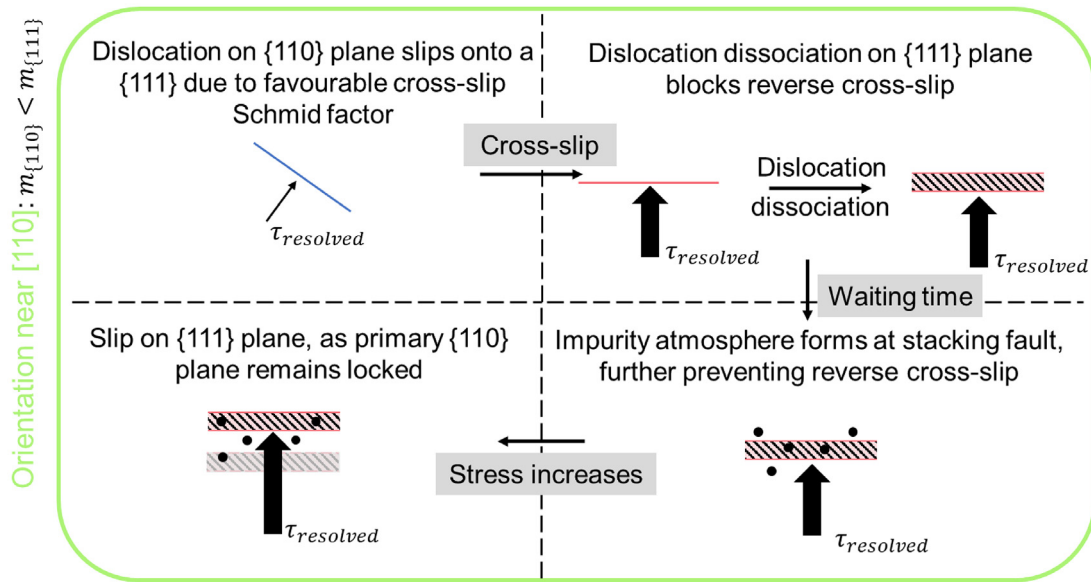


Fig. 10. Interaction of cross-slip and increased impurity mobility leading to the locking of slip on the $\{110\}$ plane and facilitating slip on $\{111\}$ planes at 400°C , low loading rates and appropriate crystallographic orientations. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

10). Even if these dissociated dislocations are able to reverse cross-slip back to their original $\{110\}$ plane, they leave behind dislocation jogs pinned by impurities, which impede dislocation motion [50,51].

4.2.3. Orientation + temperature + impurities + time: slip on $\{111\}$:

This synergetic effect effectively blocks the primary $\{110\}$ slip system as a result of diminishing mobile dislocation density and consequently, the stress in the micropillar continues to rise until it reaches the critical stress required to activate an alternative mechanism of plastic deformation. In this case, the subsidiary $\{111\}$ slip system becomes active, allowing dissociated Shockley or synchroshear partials to move in the presence of impurities, leading to a change in the active slip system (bottom left in Fig. 10).

4.3. Conditions for a pronounced yield stress anomaly:

To demonstrate that the pronounced yield stress anomaly in MnS arises only when specific combinations of crystallographic orientation, temperature, and loading rate are met simultaneously, we systematically varied these factors. Alterations diminish either cross-slip effects, impurity mobility, or both, causing the micropillar's plastic response to revert to expected behaviour:

4.3.1. Higher loading rates:

Higher loading rates reduce the time available for dislocations to cross-slip from $\{110\}$ planes to $\{111\}$ planes and for impurities to accumulate at stacking faults. This diminishes both cross-slip and impurity pinning effects, resulting in predominant slip on $\{110\}$ planes. Experimental observations confirm that at higher loading rates, the yield stress anomaly is suppressed, and deformation proceeds via conventional $\{110\}$ slip (Fig. 8). Increased serrations (cf. Fig. 5) suggest a transition to a locking-unlocking mechanism as dislocations are not fully locked on $\{111\}$ planes.

4.3.2. Lower temperatures:

At 200°C and below, impurity mobility is insufficient to pin dislocations, maintaining $\{110\}$ slip as the primary deformation mechanism. Experiments show no yield stress anomaly under these conditions (cf. Fig. 8), aligning with expected behaviour.

4.3.3. Crystallographic orientation:

The near $[100]$ orientation shows no yield stress anomaly or slip system change at 400°C , even at low loading rates. Cross-slip from $\{110\}$ to $\{111\}$ planes is less favourable due to similar Schmid factors (Table 8), preventing dislocation trapping on $\{111\}$ planes and reducing impurity pinning.

Nonetheless, at 400°C , impurities still acquire increased mobility, enabling interactions with dislocations unrelated to cross-slip jogs. Entire dislocation lines or stacking faults of dissociated dislocations can be affected and pinned. This is evident from the higher number of serrations (cf. Fig. 5) encountered in the near $[100]$ orientation at 400°C , and from the non-linear behaviour of the CRSS over temperature (Fig. 8). It underscores that in the orientation near $[110]$, the mechanism is specifically related to cross-slip, as otherwise the same slip system activation and mechanical behaviour over temperature would be expected.

At 600°C and in the $[110]$ compression direction, slip continues to take place on $\{111\}$ planes at a higher CRSS compared with $\{110\}$ critical stresses. However, the critical stresses fall on both $\{110\}$ and $\{111\}$ planes and solute mobility increases to the point of disappearing serrations, marking the approaching end of the yield stress anomaly in terms of temperature.

We believe several factors contribute to the absence of documented pronounced yield stress anomalies in MnS in the literature thus far, which are mostly related to the specific conditions that need to be fulfilled to observe it. First, the region where no yield stress anomaly is expected is limited to orientations close to $[100]$, which is the preferred orientation for testing single crystals of materials with a rock salt crystal structure. This preference arises because the $\{100\}$ planes represent cleavage planes that facilitate sample preparation [44], while testing in $\{111\}$ orientation may require suppression of cracking to activate the hard slip system [45,52]. Second, what is missing from most previous work on these effects, is an investigation of the active slip systems involved, particularly in the more limited work on MnS. Compression of polycrystals or hardness testing as a function of temperature was most commonly used to study plasticity, e.g. by Chao et al. on MnS single crystals [33], but both involve complex stress fields precluding isolated activation of hard slip systems due to the range of Schmid factors on many different slip systems. Microcompression on the other hand, as employed here, offers the opportunity to suppress cracking even in hard single crystals to identify the most prominent active slip system as well

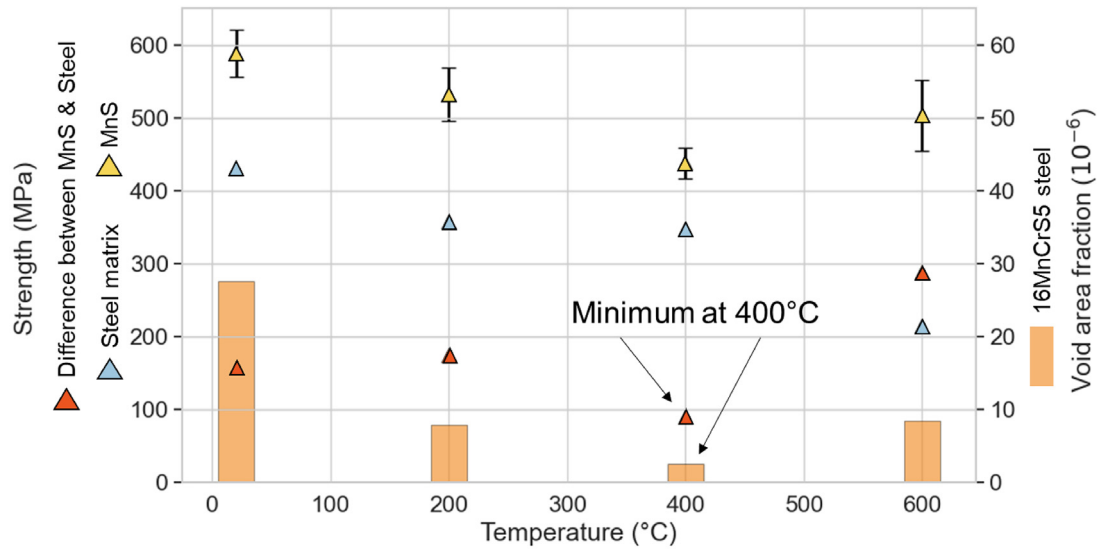


Fig. 11. The trend of the mean strength of MnS micropillars at 5% plastic strain from the area near [100] and the yield stress of 16MnCrS5 case-hardening steel over temperature are given in yellow and blue respectively. The difference between the strength of these phases over temperature is also given in red. As single tests were performed on steel, no standard deviation is given for the strength of steel and the difference between steel and MnS. The area void fraction over temperature as calculated from the bending tests is given once more in orange.

the corresponding CRSS at the same time [53]. Third, we show that the anomaly is particularly dependent on the applied loading rate, being only fully present at slow loading rates, which might have not been tested before.

4.4. Calculation of mechanical contrast and explanation of damage behaviour

Based on the presented results, it is expected that the deformation of MnS inclusions in 16MnCrS5 case-hardening steel during tensile testing is best described by the behaviour observed in the near [100] orientation, rather than by a yield stress anomaly. This arises from several factors. Firstly, MnS inclusions are often times polycrystalline [54,55] and hardness measurements of inclusions tested directly in steel have shown a linear decrease with increasing temperature [34]. Secondly, the steel microstructure surrounding the MnS inclusion entails phase and grain boundaries, leading to incompatibility issues and more complex stress states acting on the inclusions compared to the more simple uniaxial stress condition in this work. Lastly, industrially relevant forming processes exhibit a range of different deformation rates, contrasting with the low loading rate that is necessary to observe the yield stress anomaly. Having established the most probable operative mechanisms and critical stresses for deformation of MnS inclusions in the 16MnCrS5 steel, it becomes possible to estimate the mechanical contrast in the microstructure based on differences in the flow stress. For this, the yield stress of the steel (Table 2) is compared with the flow stress at 5% plastic strain at a loading rate of 0.02 mNs^{-1} of the MnS micropillars for the near [100] orientation. Their trends over the temperature as well as their differences are depicted in Fig. 11.

We compare the strength of the MnS micropillar at 5% plastic strain to the yield stress of the steel for two reasons: Firstly, the previously discussed effect of cross-slip in this orientation leads to large variations in stress during the initial stages of the compression experiment. However, by 5% plastic strain, these variations diminish considerably, as the stress is no longer dependent on whether dislocation nucleation and/or cross-slip events happen relatively early or late during the initial loading of the micropillar. Secondly and more importantly, damage starts to nucleate only after strain has already accumulated in the sample and the inclusions, as seen for example in [56], making the mechanical contrast at 5% plastic strain of MnS more valuable for the prediction of damage. Fortunately, inclusions within the steel matrix

show a similar diameter to the tested micropillars, indicating that a correction for size effect is not necessary.

The mechanical contrast in terms of flow stress changes with temperature. In particular from 200 °C to 400 °C the mechanical contrast is reduced and finds its minimum before it rises to the maximum value at 600 °C. Comparing this trend with the trend of damage prevalence over temperature Fig. 1 reveals that at least two different effects influence the damage accumulation. Firstly, the thermal activation of slip in MnS increases the ductility over the whole temperature range and strongly influences the damage prevalence from 20 °C to 200 °C. Even though the mechanical contrast remains roughly constant in this region, a decrease of damage is observed. At 400 °C however, the mechanical contrast is additionally reduced resulting in a second effect on the damage prevalence. Presumably, at this temperature MnS inclusions can keep up with the plastic deformation of the surrounding steel matrix in a more efficient way and hence initiate less damage sites during deformation. This behaviour matches the observed minimum prevalence of damage sites (Fig. 1) in the tensile test specimen at 400 °C. Most interestingly, at 600 °C the mechanical contrast plays an even larger role for damage prevalence. At this temperature, the contrast is at its maximum and corresponds to a higher prevalence of damage sites than observed at 400 °C. This is despite the fact that the promoted ductility due to thermal activation of slip systems was expected to decrease damage initiation. A third effect that might influence the stress state and hence the damage formation over temperature is the aspect of thermal expansion of inclusions and steel. In this work however, thermal expansion is assumed to be negligible, as it is similar for both materials in the observed temperature range. Liu and al showed for ferrite a thermal expansion in the terms of increasing length of roughly 1% when increasing the temperature from 400 K to 1000 K [57]. Furuseth and Kjekshus showed for MnS also an increase of roughly 1% in the same temperature range by the terms of change of lattice constant [58]. Additionally, they showed that the thermal expansion obeys a linear trend in both ferrite and MnS for the temperature range discussed in this work. This means that thermal expansion alone cannot lead to the observed minimum of the damage prevalence at 400 °C. Hence we underline once more that the mechanical contrast between two phases in a microstructure is a crucial factor in damage-controlled forming processes, and it cannot be assumed to remain constant over temperature. Understanding how to manipulate mechanical contrast with temperature expands the spectrum of parameters available for damage

control, potentially enhancing component lifespan, performance and simultaneously allowing for more effective light-weight design to reduce emissions and energy consumption. For example, based on the results of this work, when focusing solely on the damage prevalence, it is advisable to limit the temperature of forming processes with strain rates similar to the conducted tensile tests for 16MnCrS5 case-hardening steel to 400 °C. Although it needs be considered how a change of temperature influences other metallurgical phenomena, limiting forming temperatures to 400 °C impedes formation and evolution of damage sites related to MnS inclusions while simultaneously preventing the formation of damage sites that are related to damage mechanisms only active at higher temperatures.

5. Conclusion

We studied the mechanisms of plastic deformation in MnS in the range from room temperature to 600 °C and their effect on the prevalence of MnS inclusion-induced damage sites in 16MnCrS5 case-hardening steel in the same temperature range. Our results indicate that temperature plays an important role for both the plasticity of MnS and the damage prevalence initiated during forming. This highlights that damage controlled forming can be achieved by controlling the mechanical contrast between phases through temperature regulation. The main conclusions are summarised as follows:

- The dominant influence on damage prevalence shifts from softening effects at lower temperatures to increasing mechanical contrast at higher temperatures, resulting in a minimum damage prevalence at 400 °C.
- We confirmed the main slip system of MnS as $\{110\}\langle\bar{1}10\rangle$ and the activation of the $\{111\}\langle\bar{1}10\rangle$ slip system in specific orientations at elevated temperatures, using micropillar compression in conjunction with TEM analysis.
- We determined the CRSS of the $\{110\}\langle\bar{1}10\rangle$ slip system in the complete temperature range at three different loading-rates and the CRSS for the $\{111\}\langle\bar{1}10\rangle$ slip system at 400 °C and 600 °C.
- At 400 °C, an orientation-dependent and loading rate-dependent yield strength anomaly associated with a change in slip system was observed for the first time in MnS. We suggest this anomaly is related to cross-slip of dislocations from the $\{110\}$ to the $\{111\}$ plane.
- We expect the yield stress anomaly to have negligible influence on damage initiation due to its orientation and loading rate dependence and the resulting reduced effect in non-single-crystalline MnS inclusions experiencing a complex stress field during deformation.
- By considering the results of this study along with metallurgical phenomena that also influence the microstructure, we propose that it should be possible to identify a process window that minimises damage prevalence during forming.

CRediT authorship contribution statement

Maximilian A. Wollenweber: Writing – review & editing, Writing – original draft, Investigation, Data curation. **Jonas Werner:** Writing – review & editing, Investigation. **Carl F. Kusche:** Writing – review & editing, Investigation. **Chunhua Tian:** Investigation. **Pei-Ling Sun:** Writing – review & editing, Investigation. **Jannik Gerlach:** Writing – review & editing, Investigation. **Talal Al-Samman:** Writing – review & editing, Supervision, Project administration, Funding acquisition. **Sandra Korte-Kerzel:** Writing – review & editing, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors express their gratitude to Kevin Kistermann from the Central Facility for Electron Microscopy, RWTH Aachen University, Aachen, Germany for fabricating the TEM lamella. The authors also express their gratitude to the Deutsche Forschungsgemeinschaft (DFG), Germany for financial support in context of the Collaborative Research Centre CRC/Transregio 188/2 “Damage Controlled Forming Processes”, project B02, project B04 and project A08, project no. 278868966, and in the context of SFB1394 Structural and Chemical atomic Complexity – From Defect Phase Diagrams to Material Properties, project no. 409476157.

Appendix A. Supplementary data

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.actamat.2025.120987>.

Data availability

Data will be made available on request.

References

- [1] M.J. de Villafranca Casas, S. Smit, A. Nilsson, T. Kuramochi, Climate targets by major steel companies: an assessment of collective ambition and planned emission reduction measures, *Energy Clim. Chang.* 5 (2024) 100120.
- [2] I. Fărcean, G. Proștean, A. Socalici, Sustainable development indicators in the steel industry, in: *Journal of Physics: Conference Series*, Vol. 2540, IOP Publishing, 2023.
- [3] D. Leeson, N. Mac Dowell, N. Shah, C. Petit, P.S. Fennell, A techno-economic analysis and systematic review of carbon capture and storage (CCS) applied to the iron and steel, cement, oil refining and pulp and paper industries, as well as other high purity sources, *Int. J. Greenh. Gas Control.* 61 (2017) 71–84.
- [4] E. Karakaya, C. Nuur, L. Assbring, Potential transitions in the iron and steel industry in Sweden: towards a hydrogen-based future? *J. Clean. Prod.* 195 (2018) 651–663.
- [5] O. Iuorio, L. Napolano, L. Fiorino, R. Landolfo, The environmental impacts of an innovative modular lightweight steel system: The Elissa case, *J. Clean. Prod.* 238 (2019) 117905.
- [6] C. Rynkiewicz, The climate change challenge and transitions for radical changes in the European steel industry, *J. Clean. Prod.* 16 (7) (2008) 781–789.
- [7] C. Kusche, T. Reclik, M. Freund, T. Al-Samman, U. Kerzel, S. Korte-Kerzel, Large-area, high-resolution characterisation and classification of damage mechanisms in dual-phase steel using deep learning, *PloS One* 14 (5) (2019) e0216493.
- [8] O. Hering, A. Dunlap, A.E. Tekkaya, A. Aretz, A. Schwedt, Characterization of damage in forward rod extruded parts, *Int. J. Mater. Form.* 13 (2020) 1003–1014.
- [9] K. Langenfeld, L. Lingnau, J. Gerlach, P. Kurzeja, R. Gitschel, F. Walther, T. Kaiser, T. Clausmeyer, Low cycle fatigue of components manufactured by rod extrusion: Experiments and modeling, *Adv. Ind. Manuf. Eng.* 7 (2023) 100130.
- [10] S. Rosenthal, F. Maaß, M. Kamaliev, M. Hahn, S. Gies, A.E. Tekkaya, Lightweight in automotive components by forming technology, *Automot. Innov.* 3 (3) (2020) 195–209.
- [11] J.P. Hoefnagels, C.C. Tasan, F. Maresca, F.J. Peters, V.G. Kouznetsova, Retardation of plastic instability via damage-enabled microstrain delocalization, *J. Mater. Sci.* 50 (21) (2015) 6882–6897.
- [12] T. Vermeij, C. Mornout, V. Rezazadeh, J. Hoefnagels, Martensite plasticity and damage competition in dual-phase steel: A micromechanical experimental–numerical study, *Acta Mater.* 254 (2023) 119020.
- [13] C.C. Tasan, J.P. Hoefnagels, M. Diehl, D. Yan, F. Roters, D. Raabe, Strain localization and damage in dual phase steels investigated by coupled in-situ deformation experiments and crystal plasticity simulations, *Int. J. Plast.* 63 (2014) 198–210.
- [14] C.C. Tasan, M. Diehl, D. Yan, M. Bechtold, F. Roters, L. Schemmann, C. Zheng, N. Peranio, D. Ponge, M. Koyama, An overview of dual-phase steels: advances in microstructure-oriented processing and micromechanically guided design, *Annu. Rev. Mater. Res.* 45 (1) (2015) 391–431.
- [15] A. Tekkaya, P.-O. Bouchard, S. Bruschi, C. Tasan, Damage in metal forming, *CIRP Ann* 69 (2) (2020) 600–623.
- [16] M. Khelifa, M. Oudjene, A. Khennane, Fracture in sheet metal forming: Effect of ductile damage evolution, *Comput. Struct.* 85 (3–4) (2007) 205–212.
- [17] A.E. Tekkaya, N.B. Khalifa, O. Hering, R. Meys, S. Myslicki, F. Walther, Forming-induced damage and its effects on product properties, *CIRP Ann* 66 (1) (2017) 281–284.

- [18] O. Hering, A.E. Tekkaya, Damage-induced performance variations of cold forged parts, *J. Mater. Process. Technol.* 279 (2020) 116556.
- [19] M.A. Wollenweber, C.F. Kusche, T. Al-Samman, S. Korte-Kerzel, On the automated characterisation of inclusion-induced damage in 16MnCrS5 case-hardening steel, *Adv. Ind. Manuf. Eng.* 7 (2023) 100123.
- [20] L.A. Lingnau, J. Heermant, J.L. Otto, K. Donnerbauer, L.M. Sauer, L. Lückner, M. Macias Barrientos, F. Walther, Separation of damage mechanisms in full forward rod extruded case-hardening steel 16MnCrS5 using 3D image segmentation, *Materials* 17 (12) (2024) 3023.
- [21] C. Sombuthawee, S. Bonsall, F. Hummel, Phase equilibria in the systems ZnS-MnS, ZnS-CuInS₂, and MnS-CuInS₂, *J. Solid State Chem.* 25 (4) (1978) 391–399.
- [22] C.F. Kusche, J.S.-L. Gibson, M.A. Wollenweber, S. Korte-Kerzel, On the mechanical properties and deformation mechanisms of manganese sulphide inclusions, *Mater. Des.* 193 (2020) 108801.
- [23] H. Chao, L.H. Van Vlack, F. Oberlin, L. Thomassen, Inclusion Deformation: I: The Mechanism of Fracture and Deformation in MnS, Report, Department of Chemical and Metallurgical Engineering, University of Michigan, 1962.
- [24] Y. Zhou, Y. Xue, D. Chen, Y. Wang, B. Zhang, X. Ma, Atomic-scale configurations of synchrohear-induced deformation twins in the ionic MnS crystal, *Sci. Rep.* 4 (1) (2014) 5118.
- [25] F. Matsuno, S.-i. Nishikida, H. Ikesaki, Mechanical properties of manganese sulphides in the temperature range between room temperature and 1000°C, *Trans. Iron Steel Inst. Jpn.* 25 (9) (1985) 989–998.
- [26] E. Orozco, F. Agullo-Lopez, Hardening and serrated flow behaviour in the 300–500 K range for eu-doped alkali halides, *Acta Metall.* 34 (9) (1986) 1701–1709.
- [27] K.-H. Matucha, Elektronenmikroskopie von Gleitlinien auf verformtem NaCl, *Phys. Status Solidi (B)* 26 (1) (1968) 291–310.
- [28] E. Billur, Hot formed steels, *Automot. Steels* (2017) 387–411.
- [29] I. Flitta, T. Sheppard, Nature of friction in extrusion process and its effect on material flow, *Mater. Sci. Technol.* 19 (7) (2003) 837–846.
- [30] Y. Ren, W. Yang, L. Zhang, Deformation of non-metallic inclusions in steel during rolling process: a review, *ISIJ Int.* 62 (11) (2022) 2159–2171.
- [31] D. Yan, C.C. Tasan, D. Raabe, High resolution in situ mapping of microstrain and microstructure evolution reveals damage resistance criteria in dual phase steels, *Acta Mater.* 96 (2015) 399–409.
- [32] N. Uehata, M. Koyama, S. Takagi, K. Tsuzaki, Optical microscopy-based damage quantification: an example of cryogenic deformation of a dual-phase steel, *Isij Int.* 58 (1) (2018) 179–185.
- [33] H. Chao, L.H. Van Vlack, F. Oberlin, L. Thomassen, Inclusion Deformation: II: Hardness of MnS-FeS Microstructures, Tech. Rep., Department of Chemical and Metallurgical Engineering, University of Michigan, 1962.
- [34] K. Gove, J. Charles, The high-temperature hardness of various phases in steel, *Met. Technol.* 1 (1) (1974) 279–283.
- [35] F.-Y. Huang, Y.-H.F. Su, J.-C. Kuo, High-temperature deformation behavior of MnS in 1215MS steel, *Met. Mater. Int.* 24 (2018) 1333–1345.
- [36] L.-I. Staffansson, On the Mn-MnS phase diagram, *Met. Mater. Trans. B* 7 (1976) 131–134.
- [37] B. Kondori, A. Needleman, A.A. Benzerga, Discrete dislocation simulations of compression of tapered micropillars, *J. Mech. Phys. Solids* 101 (2017) 223–234.
- [38] M. Heller, J.-L. Gibson, R. Pei, S. Korte-Kerzel, Deformation of μm - and mm -sized Fe₂ 4wt bi-crystals with a high angle grain boundary at room temperature, *Acta Mater.* 194 (2020) 452–463.
- [39] J.S.-L. Gibson, R. Pei, M. Heller, S. Medghalchi, W. Luo, S. Korte-Kerzel, Finding and characterising active slip systems: a short review and tutorial with automation tools, *Materials* 14 (2) (2021) 407.
- [40] K. Momma, F. Izumi, VESTA 3 for three-dimensional visualization of crystal, volumetric and morphology data, *J. Appl. Crystallogr.* 44 (6) (2011) 1272–1276.
- [41] H. Strunk, Investigation of cross-slip events in NaCl crystals by transmission electron microscopy, *Phys. Status Solidi (A)* 28 (1) (1975) 119–126.
- [42] A. Nakamura, M. Ukita, N. Shimoda, Y. Furushima, K. Toyoura, K. Matsunaga, First-principles calculations on slip system activation in the rock salt structure: Electronic origin of ductility in silver chloride, *Phil. Mag.* 97 (16) (2017) 1281–1310.
- [43] Y. Zou, R. Spolenak, Size-dependent plasticity in micron- and submicron-sized ionic crystals, *Phil. Mag. Lett.* 93 (7) (2013) 431–438.
- [44] J. Amodeo, S. Merkel, C. Tromas, P. Carrez, S. Korte-Kerzel, P. Cordier, J. Chevalier, Dislocations and plastic deformation in MgO crystals: a review, *Crystals* 8 (6) (2018) 240.
- [45] P. Haasen, Dislocations and the plasticity of ionic crystals, *Mater. Sci. Technol.* 1 (12) (1985) 1013–1024.
- [46] T.E. Mitchell, Dislocations and mechanical properties of MgO-Al₂O₃ spinel single crystals, *J. Am. Ceram. Soc.* 82 (12) (1999) 3305–3316.
- [47] W. Johnston, Effect of impurities on the flow stress of LiF crystals, *J. Appl. Phys.* 33 (6) (1962) 2050–2058.
- [48] D. Caillard, Yield-stress anomalies and high-temperature mechanical properties of intermetallics and disordered alloys, *Mater. Sci. Eng.: A* 319 (2001) 74–83.
- [49] F. Sato, K. Sumino, The yield strength and dynamic behaviour of dislocations in MgO crystals at high temperatures, *J. Mater. Sci.* 15 (1980) 1625–1634.
- [50] A. Evans, P. Pratt, Work hardening in ionic crystals, *Phil. Mag.* 21 (173) (1970) 951–970.
- [51] E. Yu. Gutmanas, E. Nadgornyi, Dislocation motion in secondary slip planes in alkali halide crystals at room temperature, *Phys. Status Solidi (B)* 38 (2) (1970) 777–782.
- [52] S. Korte, W. Clegg, Discussion of the dependence of the effect of size on the yield stress in hard materials studied by microcompression of MgO, *Phil. Mag.* 91 (7–9) (2011) 1150–1162.
- [53] S. Korte-Kerzel, Microcompression of brittle and anisotropic crystals: recent advances and current challenges in studying plasticity in hard materials, *Mrs Commun.* 7 (2) (2017) 109–120.
- [54] X.-g. Liu, C. Wang, J.-t. Gui, Q.-q. Xiao, B.-f. Guo, Effect of MnS inclusions on deformation behavior of matrix based on in-situ experiment, *Mater. Sci. Eng.: A* 746 (2019) 239–247.
- [55] C. Wang, X.-g. Liu, J.-t. Gui, Z.-l. Du, Z.-f. Xu, B.-f. Guo, Effect of MnS inclusions on plastic deformation and fracture behavior of the steel matrix at high temperature, *Vacuum* 174 (2020) 109209.
- [56] F. Qayyum, M. Umar, V. Elagin, M. Kirschner, F. Hoffmann, S. Guk, U. Prah, Influence of non-metallic inclusions on local deformation and damage behavior of modified 16MnCrS5 steel, *Crystals* 12 (2) (2022) 281.
- [57] Y. Liu, F. Sommer, E. Mittemeijer, Abnormal austenite–ferrite transformation behaviour of pure iron, *Phil. Mag.* 84 (18) (2004) 1853–1876.
- [58] S. Furuseth, A. Kjekshus, On the properties of $\alpha\text{-MnS}$ and MnS_2 , *Acta Chem. Scand.* 19 (6) (1965) 1405–1410.

Chapter 8

Pre-print of publication 4

Ductile damage at MnS inclusions in 16MnCrS5 steel - influence of the phase environment

M.A. Wollenweber¹, T. Reclik¹, C. Reinhart¹, V. Maier-Kiener², T. Al-Samman¹, U. Kerzel¹, S. Korte-Kerzel¹

¹Institute of Physical Metallurgy and Materials Physics, RWTH Aachen University, Germany

²Department Materials Science, Montanuniversität Leoben, Austria

RWTH Publications Uploaded: 07 December 2025

Maximilian A. Wollenweber: Methodology, Formal analysis, Investigation, Writing - Original Draft

Tom Reclik: Software, Methodology, Formal analysis, Investigation, Writing - Original Draft. Clara Reinhart: Software, Formal analysis, Investigation, Writing - Review & Editing. Verena Maier-Kiener: Investigation, Writing - Review & Editing. Talal Al-Samman: Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition. Ulrich Kerzel: Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition. Sandra Korte-Kerzel: Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition.

(Online availability: RWTH-2025-10405)

Ductile damage at MnS inclusions in 16MnCrS5 steel - influence of the phase environment

Maximilian A. Wollenweber^{a,*}, Tom Reclik^{a,*}, Clara Reinhart^a, Verena Maier-Kiener^b, Talal Al-Samman^a, Ulrich Kerzel^a, Sandra Korte-Kerzel^a

^a*Institute of Physical Metallurgy and Materials Physics, RWTH Aachen University, Aachen, 52074, Germany*

^b*Department Materials Science, Montanuniversität Leoben, Leoben, 8700, Austria*

Abstract

Forming-induced damage strongly affects the service life and mechanical properties of components made from 16MnCrS5 case-hardening steel. In this study, we quantitatively investigate how the local phase environment, specifically ferritic, pearlitic, and mixed regions, around damage sites associated with manganese sulphide (MnS) inclusions affects their nucleation and growth. Using a novel approach based on machine learning-driven segmentation of high-resolution scanning electron micrographs from in-situ tensile tests, we achieve reproducible and efficient identification of phases in ferritic–pearlitic microstructures and quantification of damage sites. Our results reveal that ferritic environments lead to increased damage prevalence when compared to mixed and pearlitic environments. In particular, the growth of damage sites is markedly facilitated when the local ferrite fraction is increased. We explain this with the low strain hardening capability of ferrite compared to pearlite, determined via spherical nanoindentation. In contrast, the nucleation probability is not strongly dependent on the immediate phase environment, as the yield strength of ferrite and pearlite are similar.

Keywords: Microstructural damage, Segmentation, Scanning Electron Microscopy, Machine learning, Steel, MnS, Spherical nanoindentation

1. Introduction

Research on structural materials typically focuses on improvements of mechanical properties. In particular, when improving service life of components, the reduction of forming-induced ductile damage is of importance. Ductile damage, commonly characterised by voids within the microstructure, is known to compromise components' mechanical properties such as energy absorption during impact and endurance under cyclic loading conditions [1, 2, 3]. Consequently, maximising component performance requires minimising the prevalence of damage sites, usually quantified by their number or total area, during forming operations.

Typically, a high prevalence of damage sites is predominantly observed in materials that exhibit significant mechanical contrast between microstructural phases [4,

5, 6]. Irrespective of the exact mechanism of initiation or material system, damage formation is governed by the different mechanical properties and spatial arrangement of constituent phases. These can be inherent phases, as in dual-phase steels [7, 8, 9, 10] or magnesium alloys [11]. In these cases, the mechanical contrast between the constituent phases leads to strain partitioning and subsequent nucleation of damage sites at phase interfaces or via fracture of the more brittle constituent phase [12, 13]. In many other materials, however, damage initiates not from main constituent phases but from particles, such as inclusions or precipitates [14, 15, 16]. Whether intentionally added or formed due to impurities [17, 18], these inclusions can act as nucleation points for damage.

One example is case-hardening steel 16MnCrS5, which exhibits damage sites that are mainly attributed to the fracture of manganese sulphide (MnS) inclusions or delamination at their interfaces [19]. The damage behaviour and its negative impact on the mechanical properties of this steel are well described in the literature and generally attributed to the different mechanical proper-

*These authors contributed equally

Email addresses: wollenweber@imm.rwth-aachen.de (Maximilian A. Wollenweber), reclik@imm.rwth-aachen.de (Tom Reclik)

ties of MnS and the steel matrix [20, 21, 22, 23]. However, addition of Manganese and subsequent formation of MnS also provides benefits for manufacturing processes such as resistance to hot shortness [24, 25].

In earlier work, we have shown that both the size and number of damage sites differ between individual MnS inclusions and depend on each inclusion’s specific location within the steel microstructure [19]. Notably, this variation occurs even at identical levels of applied global strain. We hypothesise that this heterogeneous distribution of damage sites is partially driven by variations in local phase composition (ferrite vs. pearlite), surrounding each MnS inclusion. Unlike dual-phase steels, where interactions between two inherent constituent phases primarily govern damage formation, the heterogeneous microstructure of 16MnCrS5 contains three distinct phases: ferrite, pearlite and the inclusion phase MnS. As a result, more complex interactions contribute to damage formation. The significance of MnS position in relation to damage has also been recognised by other researchers evaluating individual inclusions [26, 27]. However, no quantitative framework directly linking an inclusion’s local microstructural environment to its propensity for nucleating and growing damage has yet been established. Developing such a framework is crucial to gain a deeper understanding of the mechanisms governing damage evolution in these multiphase alloys.

In this study we address this gap by investigating how local phase environments affect initiation and growth of inclusion-induced damage sites in multiphase microstructures. In particular, we propose that ferritic regions, being softer than pearlite and accommodating greater strain, facilitate increased damage prevalence around MnS inclusions. Our central scientific question is: Does a higher proportion of ferrite adjacent to MnS inclusions lead to greater damage? Furthermore: Is this effect manifested through more frequent nucleation events, accelerated void growth, or both?

To answer these questions, we quantitatively assess how ferritic, pearlitic, and mixed-phase environments influence the number fraction, area fraction and mean size of voids associated with MnS inclusions. Our analysis provides information at both macroscopic (mm) and microscopic (μm) scales, focusing on statistical trends as well as individual MnS inclusions and their associated damage sites. We achieved this by acquiring high-resolution panoramic scanning electron microscopy (SEM) images at different strains during an in-situ tensile test of an etched 16MnCrS5 case-hardening steel sample. For subsequent automated evaluation across large image datasets, we trained two neural net-

works based on the nnU-Net architecture [28], enabling segmentation into ferrite, pearlite, MnS inclusions, and damage sites. This approach extends time-efficient statistical analysis previously applied to unetched samples [29, 30, 31, 32, 19] by incorporating all constituent phases of 16MnCrS5 (see Figure 1). Finally, to complement our statistical findings, we interpret observed trends using mechanical property measurements obtained via spherical nanoindentation within individual phases.

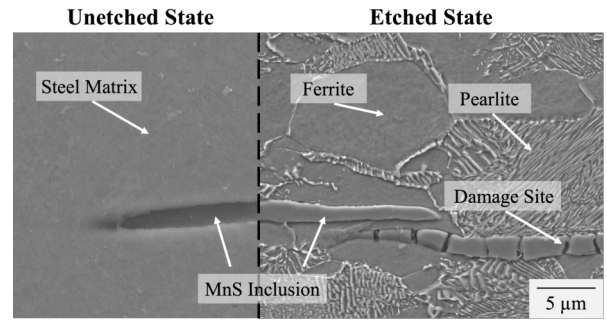


Figure 1: MnS inclusion in 16MnCrS5 case-hardening steel, showing constituent phases in the unetched (left) and etched (right) states.

2. Materials and Methods

2.1. Case-hardening 16MnCrS5 steel

Microstructure segmentation and damage quantification were conducted on a case-hardening 16MnCrS5 steel that had been produced by continuous casting. The chemical composition of the steel, previously determined in [33], is provided again in Table 1.

Its typical ferritic–pearlitic microstructure (visible only after etching) is shown in Figure 1. MnS inclusions are elongated due to the continuous casting process and embedded in the steel matrix without obvious preferences for pearlitic or ferritic interfaces. MnS inclusions can reach lengths of several tens of micrometres but typically have diameters up to $2\ \mu\text{m}$ (Figure 1). When tensile loading is applied parallel to the long axis of the MnS inclusions at ambient temperature, these inclusions typically fracture, resulting in the nucleation of damage sites. This damage initiation mechanism physically separates segments of an MnS inclusion as elastic strain is relieved; continued deformation separates the segments further, as illustrated in Figure 1. All experiments were performed on the as-received material. Aside from the in-situ tensile tests themselves, no additional forming steps or heat treatments, including case hardening, were carried out.

Table 1: Chemical composition of 16MnCrS5 case-hardening steel (in wt.%), from [33].

Element	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Co	Cu	Nb	V	Fe
Wt. %	0.20	0.17	1.24	0.03	0.03	1.05	0.04	0.14	0.03	0.01	0.16	0.01	0.01	Bal.

2.2. Sample preparation

To prepare samples for in-situ tests, dog-bone-shaped specimens were machined from a 16MnCrS5 steel billet using electrical discharge machining. The sample geometry (Figure 2) featured a sheet thickness $h = 1.5$ mm, gauge length $l = 3.56$ mm, and gauge width $w = 1.5$ mm.

Once machined, the samples were metallographically prepared to obtain a flat surface suitable for SEM imaging. To this end, each sample was initially ground with silicon carbide (SiC) sandpaper up to a grit size of 4000 and subsequently polished mechanically with water-based diamond suspensions ($3\mu\text{m}$ and subsequently $1\mu\text{m}$) and water as a lubricant. As a final polishing step, the sample surface was polished with an oxide polishing suspension (OPS), followed by etching in 3 % nital for 9 s to reveal the pearlitic phase.

2.3. In-situ tensile test

After mechanical preparation, the sample was mounted in the chamber of the SEM (Clara, Tescan) and the surface was imaged using panoramic imaging with a secondary electron (SE) detector at an acceleration voltage of 10 kV and a beam current of 1 nA. In total, 150 images were acquired per strain state, each covering an area of $100\mu\text{m} \times 100\mu\text{m}$, at a resolution of $100\mu\text{m}/3072\text{px}$ and an overlap of 15 % on each side. These images were subsequently stitched to generate a single panoramic image encompassing approximately 1.1 mm^2 . Following initial imaging of the sample surface, the sample was transferred to a micro-tensile stage (Proxima, microMECHA) and deformed incrementally to a plastic strain of 3 %. After each deformation step, the same surface region was re-imaged using the panoramic SEM approach. This procedure was repeated at further plastic strains of 4 more times at plastic strains of 5 %, 8 %, 10 % and 14 %. To compensate for specimen elongation during deformation and to maintain consistent coverage of the chosen region at increasing strain levels, the number of images per panoramic scan was gradually increased up to 180 images at the highest strain level. This adjustment resulted in a total imaged area of about 1.3 mm^2 at (14 %) final strain.

2.4. Image segmentation using neural networks

To enable segmentation of the microstructure into its constituent phases and to quantify damage sites, two separate neural networks, both based on the nnU-Net architecture [28], were trained and fine-tuned using the acquired SEM data. Hereafter, these networks are referred to as phase-net and damage-net, according to their respective purposes. In total, three different datasets were prepared for training purposes and annotated using the Computer Vision Annotation Tool (CVAT) [34]. The characteristics of these datasets are summarised in Table 2 and more information on the design choices of the neural networks is given in the supplementary materials.

During in-situ testing, plastic deformation is visible on the surface as slip steps, which are considered as artefacts in the context of microstructure segmentation. Thus, to ensure consistent segmentation accuracy across all strain levels, the training dataset included images with both low and high densities of surface artefacts. To this end, images were randomly selected from those acquired at strains of 3 % and 10 % to represent a broad range of deformation states. For dataset 1, a total of 70 images were manually labelled for microstructural constituents and used as ground truth. This dataset was used both for the training of phase-net and the pre-training of damage-net. Dataset 2 comprised 119 manually labelled images, reflecting the fact that damage sites occupy only a small fraction of each image. During network training and evaluation, 80 % of each dataset was allocated to training/validation, while the remaining 20 % was reserved for testing. After training, phase-net segments the microstructure into distinct classes: "Ferrite", "Pearlite" and "MnS", directly corresponding to microstructural features shown in Figure 1. Damage-net, on the other hand, was specifically trained to identify damage sites and assigns a single class labelled "Damage". The output from damage-net enables localisation of all present damage sites as well as quantitative assessment including total void area and number of individual sites, required for evaluating damage prevalence.. By combining spatial information provided by both phase-net and damage-net outputs, it becomes possible to determine the local microstructural environment surrounding each MnS inclusion and associated

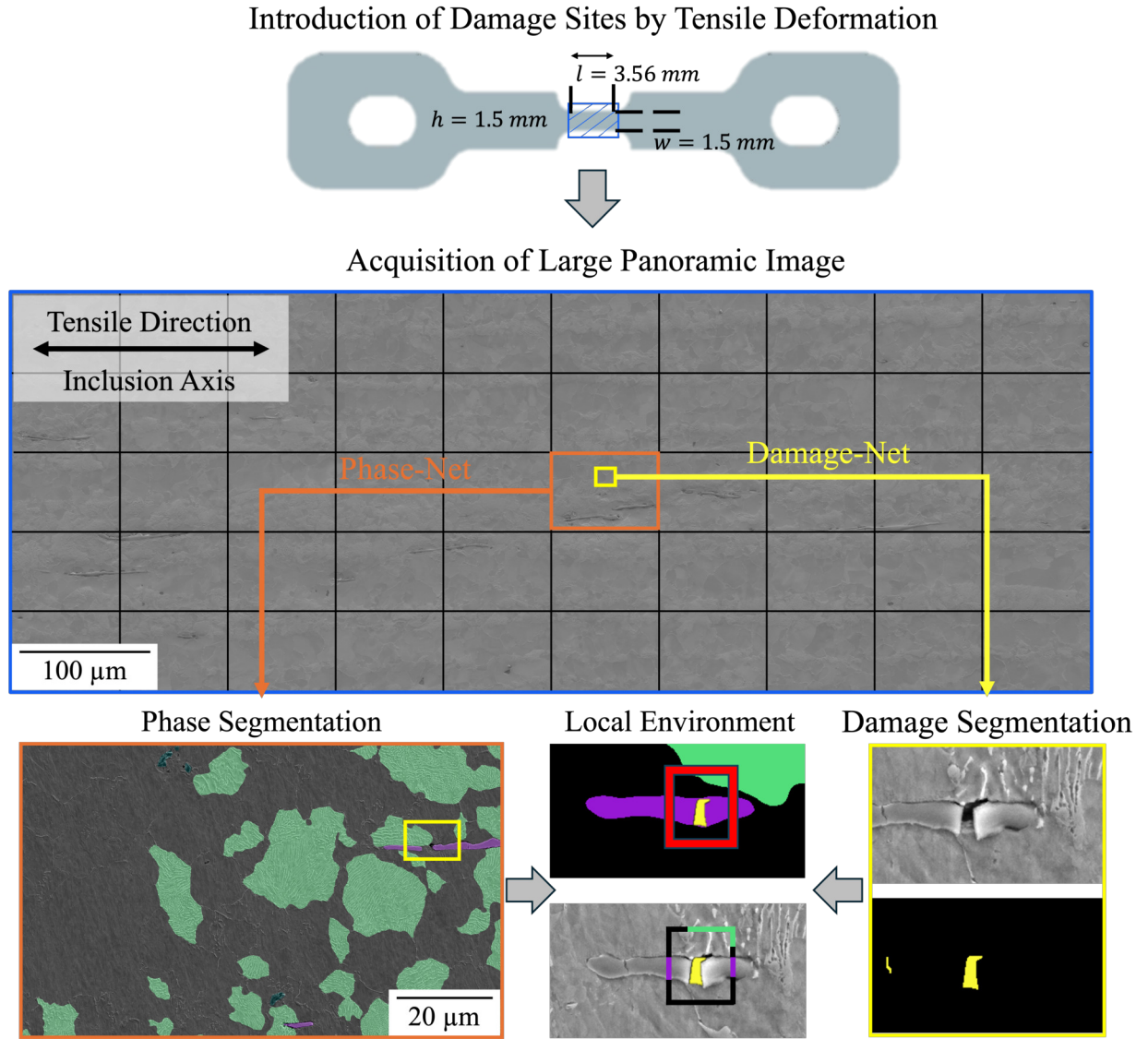


Figure 2: Workflow for determining the influence of local phase environments on damage behaviour in 16MnCrS5 steel. From top to bottom: samples are machined, deformed incrementally via interrupted tensile tests, and imaged after each step using high-resolution panoramic SEM. The resulting images are segmented by two neural networks, one for microstructural phases (orange arrows) and one for damage sites (yellow arrows). Combining these results enables evaluation of the local microstructural environment surrounding each damage site.

Table 2: Summary of datasets used to train both neural networks.

Dataset	deformation mode	number of images	classes	networks
Set 1	tensile tested	70	Ferrite, Pearlite, MnS, artifact	fine-tuning phase-net, pre-training damage-net
Set 2	tensile tested	119	Damage	fine-tuning damage-net

damage site. Importantly, both networks operate simultaneously on the same captured SE images, as illustrated in Figure 2, producing independent segmentation results. Further information on network design decisions and their reasoning can be found in the supplementary material.

2.5. Determination of local environment

Once the positions of both damage sites and microstructural constituents have been identified through segmentation, the local environment surrounding each individual damage site is determined. For this purpose, two rectangular regions are constructed above and below each damage site. To capture damage site environment accurately, the dimensions of each rectangle are scaled according to the size of its respective damage site. Rectangular shapes were chosen to enable evaluation parallel to the tensile direction and to simplify analysis. In order to construct both rectangles, first their horizontal boundaries are set in a way that the left edge begins 20 pixels to the left of the leftmost point of the damage site and the right edge ends 20 pixels to its rightmost point. Similarly, in height, the lower boundary of the upper rectangle is set 20 pixels above the uppermost point of each damage site, while the upper edge of the lower rectangle is set 20 pixels below the lowest point of each damage site. The thickness of each rectangle is consistently set at 16 pixels. These dimensions ensure that labelling artefacts at MnS interfaces are excluded from analysis and that representative areas from local microstructures are evaluated. An example of the construction, along with its dimensions, is illustrated in Figure 3 a and b. The image provided is showing an MnS inclusion, its associated damage sites, and the surrounding microstructural environment after segmentation. The colours represent different segmentation classes: purple indicates MnS inclusions, green denotes pearlite, black represents ferrite, and yellow marks damage sites.

These rectangles provide a quantitative assessment of the local microstructural environment by calculating the proportion of each phase as a percentage of total pixels within the region. However, to simplify subsequent analysis, the environments surrounding damage sites are categorised into three distinct types: *ferritic*, *pearlitic* and *mixed* environment, which are also depicted schematically in Figure 3. Each rectangle is analysed individually to determine its phase composition. If more than 50 % of the pixels are identified as pearlite, the region is designated as pearlitic.

Beyond classifying regions around damage sites, this approach is also applied to areas adjacent to all MnS in-

clusions. For this purpose, two additional rectangles are constructed for each inclusion, each with a fixed width of 92 pixels and a thickness of 16 pixels. The rectangles extend from 20 pixels below the lowest point to 20 pixels above the highest point of each MnS inclusion. To evaluate phase distribution along the inclusion length, these regions are shifted in 10 pixel increments Figure 3 c–e. Note that some regions may overlap with previously assessed damage site environments Figure 3 d.

2.6. Spherical nanoindentation

To complement the SEM-based statistical analysis with local mechanical properties, spherical nanoindentation was performed on ferrite, pearlite and MnS. For this purpose, a disc-shaped sample was mounted in a Keysight G200 nanoindenter with an integrated continuous stiffness measurement (CSM) unit and a spherical tip with a nominal radius of 10 μm . Indentations in ferrite and pearlite were carried out on the 16Mn-CrS5 case-hardening steel, while MnS inclusions were probed in a X30MnAl17-1 steel, which contains larger MnS inclusions suitable for confinement of the plastic zone within a single inclusion. Tip calibration, measurement procedures, and data analysis followed the methodology described in [35]. Due to the non-self-similar geometry of the spherical tip, and a previously determined function for the constraint factor, the recorded load and depth curves were converted to equivalent stress-strain curves. The yield strength was determined via the $Rp_{0.5}$ offset method based on these curves. Elastic moduli were extracted from CSM data. In total, 7 indents were performed in ferrite, 5 indents in pearlite and 11 indents in MnS. All indents were conducted at a strain rate of 0.001 s^{-1} . Indents in MnS were conducted to a depth of 500 nm for MnS while indents in ferrite and pearlite were conducted to a depth of 1000 nm.

3. Results

3.1. In-situ observation of MnS inclusions under strain

The in-situ experiments allow us to track the mechanisms of damage nucleation and growth, changes in MnS inclusions, and their associated damage sites as a function of strain. This progression is shown using the example of one MnS inclusion in Figure 4.

The selected inclusion is representative in size and exhibits variation in its local microstructural environment along its length, which is most apparent at the initial stage but highlighted once more at a strain level of

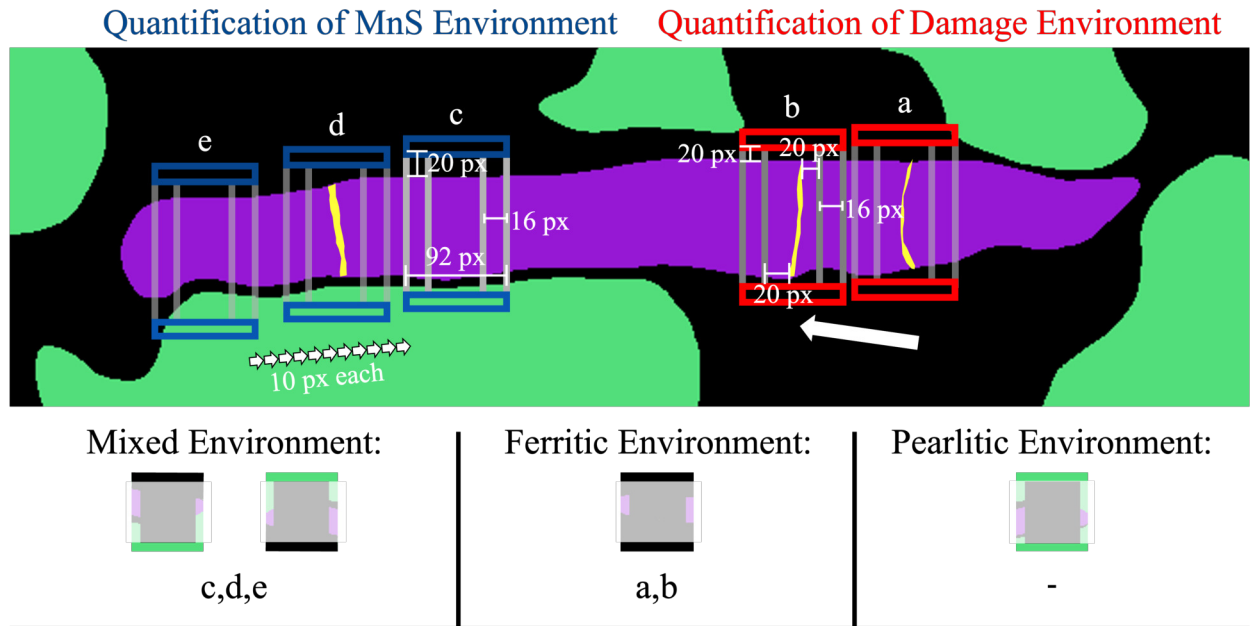


Figure 3: Visualization of the workflow to quantify microstructural environment around detected damage sites (a, b) and all possible environments (c-e), which includes environments around detected damage sites as well (d). Arrows visualize step size between recorded windows in both processes. Schematic representations below show the three environmental classes: mixed, ferritic, and pearlitic environments; green indicates pearlite, black indicates ferrite, purple indicates MnS inclusions. The non-transparent pixels are considered for the calculation of phase environment.

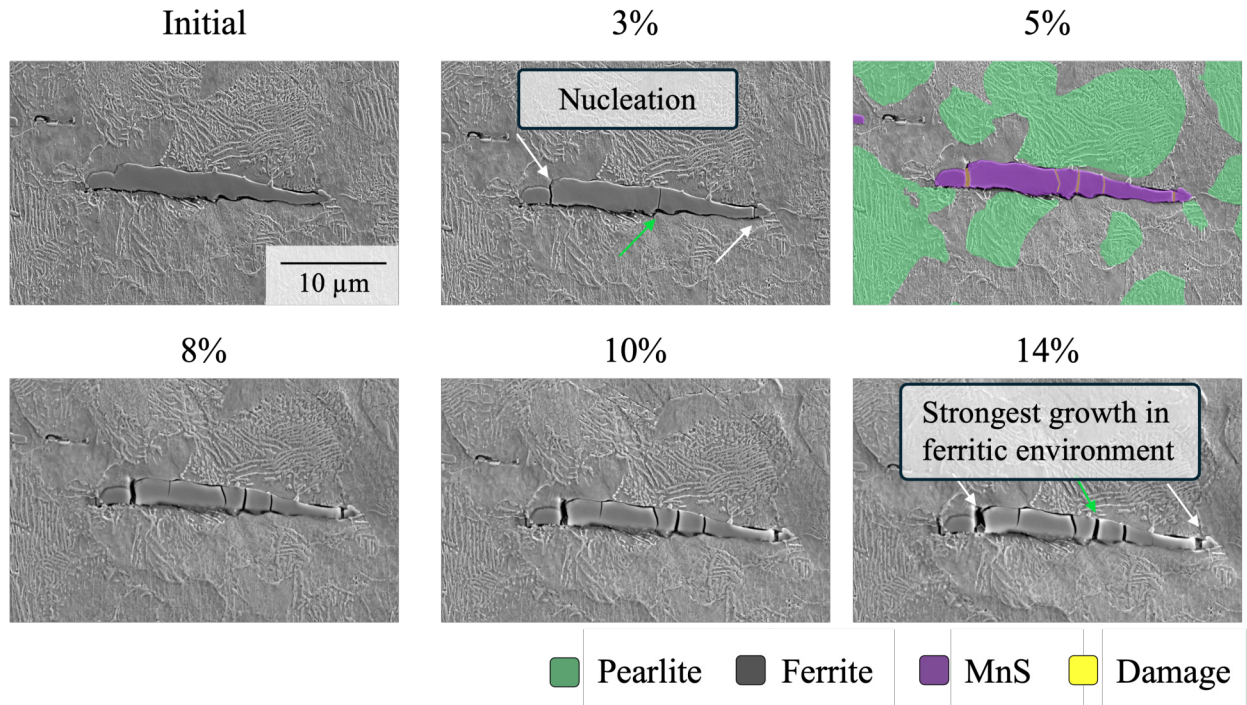


Figure 4: Sequential micrographs illustrating morphological changes in an MnS inclusion and associated damage sites during incremental straining. White arrows indicate damage sites in a ferritic environment, while green arrows indicate damage in a mixed environment. Phase information is superimposed on the microstructure in the micrograph at a strain level of 5%.

5 %. Upon deformation to 3 % plastic strain, three distinct damage sites are observed to have nucleated during this initial forming step. There is no clear preference regarding the local environment for these nucleation events: two sites formed within ferritic environment while one emerged within a mixed-phase environment. As strain increases, pre-existing damage sites expand further in width and additional new sites continue to nucleate along the inclusion. However, at a final strain of 14 %, comparison reveals that the widths of these initial three damage sites did not increase uniformly. Specifically, the damage sites situated within ferritic environments (highlighted with white arrows) grew larger than the site nucleated in a mixed-phase region (highlighted with a green arrow). To confirm these anecdotal observations, we systematically investigated the influence of the local microstructural environment on both nucleation and growth of MnS-associated damage sites using statistical analysis based on data generated by the phase-net and damage-net networks.

3.2. Evaluation of image segmentation performance

To ensure transparency regarding the reliability of our scientific findings, we begin by evaluating the performance of the segmentation networks. The output of the image segmentation process consists of masks in which each pixel is assigned to a specific class or to the matrix. We assess the quality of this classification both qualitatively and quantitatively.

For qualitative assessment, each class is assigned a distinct colour, and the resulting mask is overlaid on the original SEM image. This visual approach allows for intuitive evaluation of segmentation accuracy. A direct comparison between an SEM micrograph and its corresponding segmentation mask is presented in [Figure 5](#), facilitating a straightforward visual appraisal of both neural networks' performance.

In addition to this qualitative assessment, network performance was quantitatively evaluated using the Dice score [36], a widely-used metric for measuring accuracy in image segmentation tasks [37, 38]. Accordingly, Dice scores obtained from 14 phase segmented and 24 damage segmented images are summarised as box plots for each segmentation class in [Figure 6](#).

The box plots reveal that the medians for all classes segmented by phase-net exceed 0.9, as shown by the lines within each box. Notably, overall Dice scores as well as those for ferrite and pearlite approach 1 with only small variability across images. Although MnS exhibits a higher median Dice score than pearlite, its distribution displays greater spread. One outlier, shown as an open circle at zero results due to misclassification

at a small site. In this instance, MnS was predicted but not present in the ground truth. Such pronounced deviations occur because even minor discrepancies greatly affect metrics when site size is small.

A similar pattern is observed in the damage-net results. Here, the Dice scores display a broader variability and occasionally include low or zero values. These low scores typically arise when ground truth damage sites are not identified by the network. Examples of such cases are given in the supplementary materials.

3.3. Damage quantification during in-situ testing

Image segmentation by damage-net enable automated quantification of damage parameters by determining both the number and total area of detected damage sites. These values are normalised to the total MnS area identified by phase-net. By monitoring these parameters with increasing plastic strain, the evolution of damage sites during deformation can be characterised, as shown in [Figure 7](#). Both the number and total area of detected damage sites increase steadily with rising strain. At the final strain level observed, more than 0.2 sites per μm^2 of MnS are observed, covering approximately 4 % of the area covered by MnS.

3.4. Influence of local microstructure on damage initiation and growth

Distribution of microstructural classes around MnS inclusions. To elucidate how the local microstructural environment affects damage prevalence metrics, both area fraction and number of detected damage sites were analysed with respect to their surrounding environment. As previously described, environments were categorised into three distinct classes (fully ferritic, fully pearlitic, mixed) ([subsection 2.5](#)). Initially, we assessed the overall distribution of environmental classes surrounding MnS inclusions across all strains. The results are presented in [Table 3](#).

Table 3: Prevalence of environment classes around MnS inclusions

Class	Occurrence (%)
Fully ferritic	59 ± 2
Fully pearlitic	13 ± 1
Mixed	28 ± 1

This analysis shows that MnS inclusions are most frequently found within ferritic regions rather than mixed ones. Purely pearlitic surroundings are comparatively rare. Small deviations across different strain levels, as indicated by the standard deviation, are likely due to

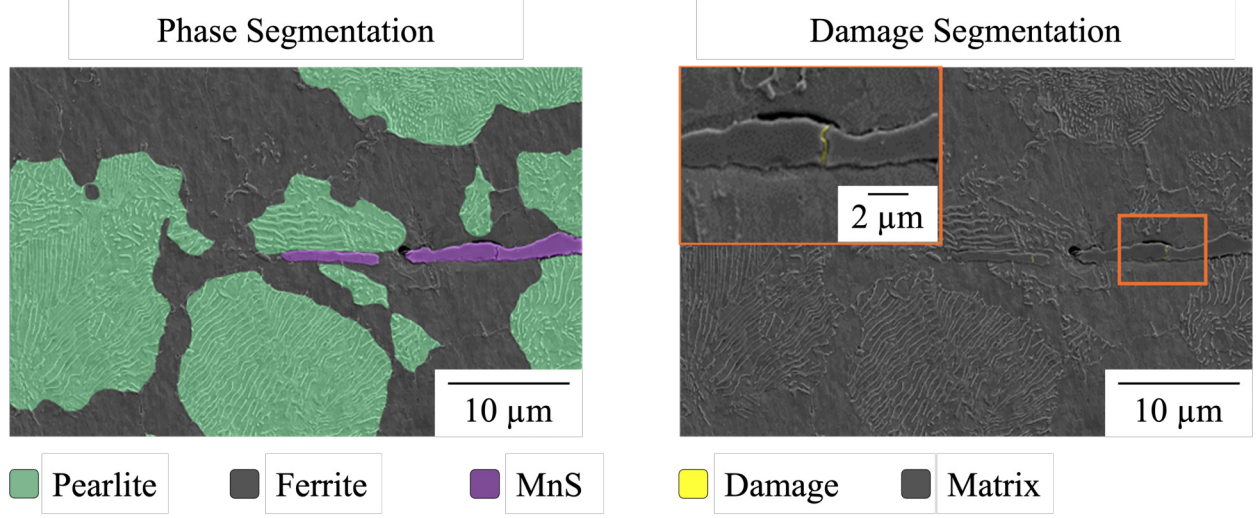


Figure 5: Secondary electron micrograph of an etched 16MnCrS5 specimen with superimposed segmentation masks: a) phase segmentation, where green denotes pearlite, purple indicates MnS, and grey corresponds to ferrite; b) damage segmentation, where yellow highlights areas classified as damage and grey represents all other areas. The inset displays a higher magnification of the rectangular region marked in orange.

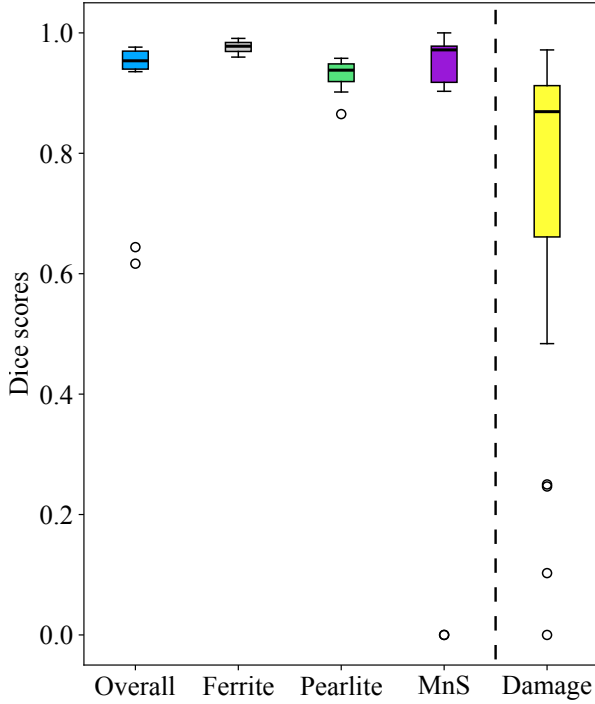


Figure 6: Box plots showing Dice scores for network performance evaluation across segmentation classes: ferrite (grey), pearlite (green), MnS inclusions (purple), overall performance for phase-net (blue), and performance for damage-net segmenting damaged regions (yellow).

small changes of the imaged area resulting from applied strain.

Environment dependence of damage nucleation. In order to unravel whether damage site nucleation preferentially occurs within specific environments, we first started by analysing damage site number fraction per class (Figure 8 a).

The data reveal that damage site nucleation is most frequent within ferritic environments compared with mixed or purely pearlitic ones and show little variation with increasing strain. On average, approximately 60 % of damage sites occur within purely ferritic surroundings, about 30 % within mixed environments and roughly 15 % within exclusively pearlitic environments. Furthermore, these results show that, although new damage sites are initiated at all strain levels (cf. Figure 4 and Figure 7), these damage sites nucleate within all environmental classes with consistent probability at any given level of applied strain. To quantitatively assess preferred nucleation sites accounting for environmental occurrence rates (Table 3), we calculated the conditional probability that a site initiates within a particular environment $p(D|E)$ using Bayes' theorem [39]

$$p(D|E) = \frac{p(E|D)p(D)}{p(E)} \quad (1)$$

where $p(E|D)$ denotes the probability for observing a certain environment at a damage site, $p(D)$ is overall

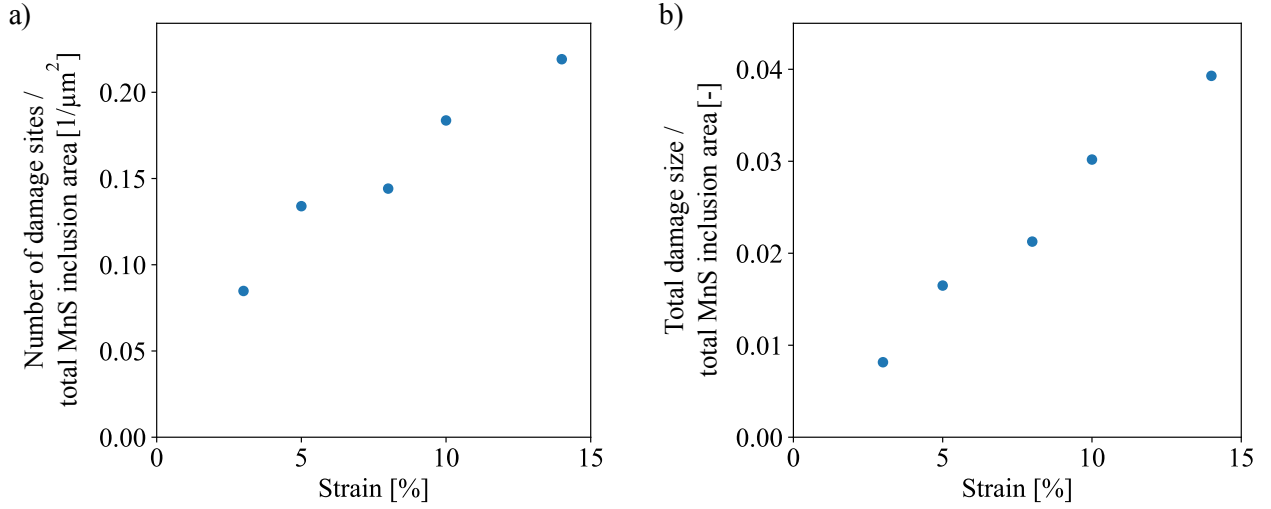


Figure 7: Evolution of key damage parameters during in-situ tensile testing as functions of strain: panel a) displays the number count, while panel b) shows total damage area, both normalised by total MnS inclusion area identified via image segmentation.

probability for damage formation, and $p(E)$ is the occurrence rate for each environmental class. $p(D|E)$ was computed per class across all strains and is visualised in Figure 8 b.

The results indicate that for all environmental classes, the likelihood of newly formed damage sites increases consistently with rising strain (except at 8 % for the mixed and pearlitic environments). However, no consistent preference emerges regarding which environment favours nucleation across all strains. For example, at 3 %, 5 %, and 10 % strains, pearlitic environments display the highest probability of damage nucleation, while at 8 % and 14 % strains ferritic regions seems more favourable.

Environment dependence of damage growth. To investigate how microstructural environment influences subsequent growth behaviour, we correlated damage area fractions with environmental class using our established method (subsection 2.5). Mean area fractions, normalised by total imaged MnS area, are displayed for each class and strain level in Figure 8 c.

An increasing trend with strain is evident for all environmental classes containing ferrite. Only within purely pearlitic surroundings between strains of 5 % and 8 % no clear trend emerges. Furthermore, there is a marked dependence of damage area on environmental class. These differences become more pronounced at higher strains, with ferritic regions exhibiting larger average damage areas than mixed or purely pearlitic regions. However, trends observed in area or number fractions may be affected by uneven distributions of environ-

ments around MnS inclusions, thus they do not directly translate to intrinsic growth rates.

Further insight was gained by analysing mean damage site size as a function of local pearlite fraction (rather than discrete classification), shown in (Figure 8 d). This approach reduces sensitivity to class imbalance effects. Here two key findings emerge: (1) At maximum strain of 14 %, higher local pearlite fraction corresponds to smaller mean void sizes. (2) When tracking changes in mean void size across strains for each bin (Figure 8 d), greater pearlite content yields less variation between strains compared to lower fractions.

3.5. Spherical indentation

Equivalent indentation stress-strain curves obtained from spherical indentation experiments, along with the corresponding constraint factor versus strain are shown in Figure 9 for ferrite, pearlite, and MnS.

Although an initial elastic regime is evident, its slope in the equivalent indentation stress-strain curve is not suitable for accurate determination of elastic modulus (E). This is because the depth in this initial regime is only a few nm and the elastic-plastic transition introduces inaccuracies in tip calibration as well as in the conversion from load-depth to stress-strain. We therefore report elastic moduli from continuous stiffness measurements (CSM), which provide higher fidelity values (Table 4). The average moduli determined fall within the ranges reported in literature for ferrite and pearlite [40, 41] and for MnS [20].

Although the elastic region in Figure 9 is not suitable for determining the elastic modulus, it can be used

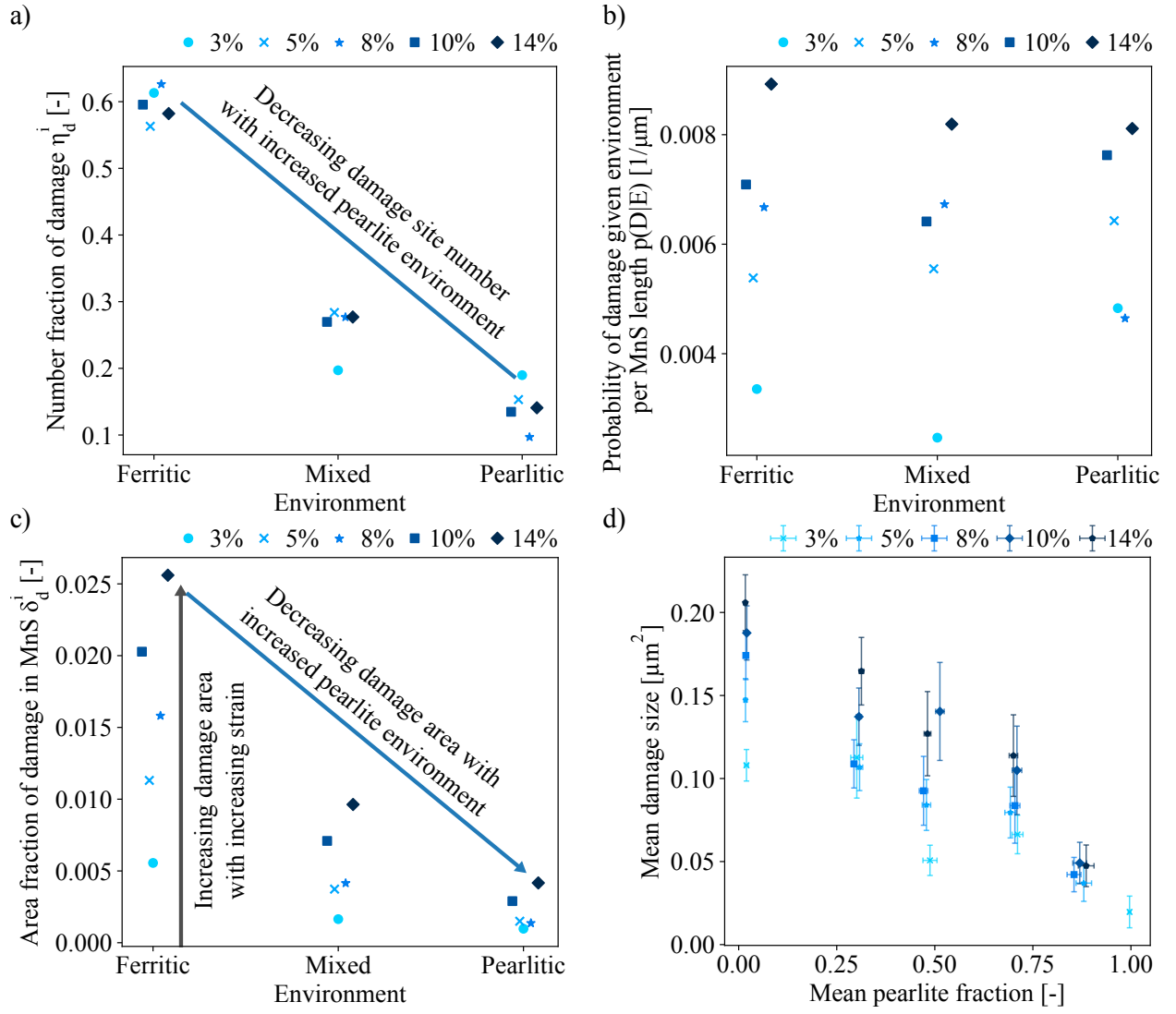


Figure 8: Damage prevalence and damage site metrics as a function of local microstructural environment and strain level. a) Number fraction of damage sites, b) probability of damage site initiation, c) mean area fraction of damage sites, d) mean size of individual damage sites. Different colours correspond to different strain levels.

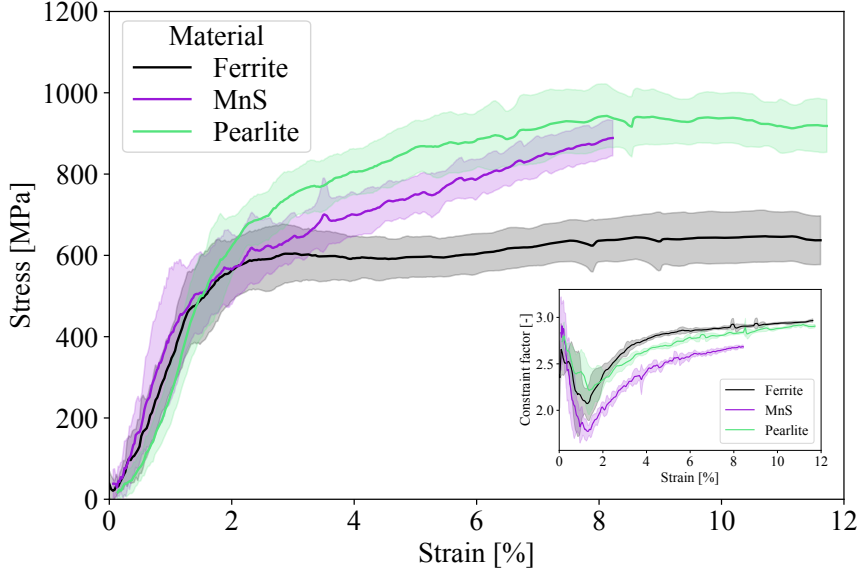


Figure 9: Equivalent stress–strain curves from spherical indentation for ferrite, pearlite, and MnS. The inset shows the constraint factor as a function of strain. Solid lines represent the mean value across multiple indents, shaded band indicate ± 1 standard deviation.

Table 4: Average elastic modulus and yield strength $Rp_{0.5}$ (mean + standard deviation) for ferrite, pearlite, and MnS. Elastic modulus determined via CSM during spherical indentation.

Phase	Ferrite	Pearlite	MnS
E (GPa)	193 ± 3	201 ± 2	111 ± 1
$Rp_{0.5}$ (MPa)	580 ± 80	720 ± 90	580 ± 60

to identify the onset of plasticity and thus define the yield strength. Here, yield strength was defined as the equivalent indentation stress at 0.5 % plastic strain using an offset based on the initial elastic slope of the stress-strain curve. The mean values for each phase are reported in Table 4. The curves show similar yield strengths for ferrite and MnS and a slightly higher value for pearlite. Although factors, such as indentation size effects, interlamellar spacing and chemistry are expected to influence the yield strength, our reported values are consistent with literature ranges for pearlite [42] as well as for MnS and ferrite [23]. Differences in plastic behaviour are apparent in the strain hardening behaviour among phases: ferrite shows modest strain hardening, pearlite shows stronger initial hardening followed by a plateau, whereas MnS displays a linear hardening behaviour at a near-constant rate throughout the measured range.

4. Discussion

4.1. Deep learning-based segmentation of phases and damage

The automated segmentation approach employing nnU-Net architecture demonstrated varying performance across different microstructural classes. While phase segmentation achieved consistently high Dice scores, damage segmentation exhibited greater variability. This disparity can be attributed to three primary factors.

Firstly, damage only constitutes 6.9×10^{-5} of the total imaged area. This severe class imbalance results in few positive training examples for the damage network. Additionally, individual damage sites are also small, with a high boundary-to-interior pixel ratio, making them especially susceptible to segmentation errors, as this characteristic is challenging for the nnU-Net’s design philosophy, which relies on learning multi-scale representations [28].

Secondly, the Dice coefficient exhibits heightened sensitivity to segmentation errors when applied to rare, small objects. A single missed damage site, even if it comprises only a few pixels, results in a Dice score of zero for that image. In contrast, misclassifying a comparable number of pixels at phase boundaries between ferrite and pearlite has negligible impact on phase segmentation scores due to the large total area of correctly classified phase pixels. This metric behaviour creates sta-

tistical instability: with few damage sites per image, the Dice score lacks sufficient sampling to stabilize, effectively computing statistics on small counts where individual errors dominate the result. Consequently, the observed variability in damage segmentation Dice scores partially reflects limitations of the metric itself rather than systematic network failure.

Lastly, distinguishing between actual damage and artefacts is particularly difficult near MnS interfaces. These artefacts can arise due to locally increased etching, scratches from metallographic preparation or even changes in surface topology during the in-situ experiment due to dislocation activity. Although unfavourable towards accurate damage segmentation, these classes of artefacts are commonly expected for high-throughput methods, in particular for methods that take large areas into account.

Despite these limitations, automated segmentation enabled analysis on a scale that is infeasible by manual annotation. Processing thousands of micrographs across multiple strain levels and environmental conditions required consistent labelling criteria that human annotators struggle to maintain over extended periods. This consistency, however, is particularly valuable for comparative studies where subtle differences in damage evolution must be quantified. While the segmentation accuracy for damage sites shows greater variability than for phase segmentation, the achieved performance is sufficient for drawing reliable conclusions about damage evolution trends and environmental effects as visualised by the consistent trends shown in Figure 8. Thus, despite these methodological considerations, clear trends emerge from our data regarding how local microstructural environment affects both damage nucleation and growth. Future work could consist of dedicated networks, with architectures tailored to small damage sites, or a two-step approach, consisting of detection and a subsequent segmentation.

4.2. Influence of local environment on damage nucleation and growth

It is widely recognised that damage prevalence in multiphase steels is primarily influenced by the strain incompatibility between a compliant and/or soft phase, which can accommodate comparatively large amounts of strain, and a stiff and/or hard phase which can accommodate comparatively less strain [43]. In the case of 16MnCrS5 damage occurs predominately in and around the MnS inclusions [44, 23], either via MnS fracture under the present loading or via interface decohesion under transverse loading [19]. Among the three constituent phases, ferrite accommodates the largest plas-

tic strain [27]. It is therefore reasonable to expect ferritic surroundings to be associated with higher damage prevalence. What remains to be unravelled is whether the environment primarily affects damage nucleation, growth, or both.

Based on the statistical results presented in Figure 8 b and d along with the in-situ observations shown in Figure 4, we separate the roles of damage nucleation and growth as a function of the local environment.

Damage nucleation. The statistical analysis of damage nucleation presented in Figure 8 b shows no consistent preference for nucleation in a particular environment across strains. The apparent fluctuations that appear to favour pearlite or ferrite at different strains are likely consistent with sampling variability rather than a physical effect since there is no mechanistic reason for the preferred environment to oscillate with strain. This is also reflected in Figure 4, where multiple nucleation events occur over a range of global strains without a consistent environmental bias.

This lack of a clear trend suggests that mechanisms beyond the surrounding environment have a larger impact on nucleation behaviour. Likely contributions include crystallographic grain orientation near inclusions and local stress concentrations arising from abrupt changes in inclusion thickness or morphology.

Damage growth. In contrast to nucleation, analysis of mean void size as a function of microstructural environment reveals a clear trend towards greater damage site growth in ferritic environments. As illustrated by Figure 4, among the three initial damage sites (white arrows), those embedded in purely ferritic neighbourhoods expand most with increasing strain. Our statistical analysis based on image segmentation (Figure 8) d confirms this stronger growth of damage sites in ferritic environments. Consistent with this observation, the resulting smaller mean void size in the vicinity of pearlite is particularly pronounced where the pearlite fraction is highest. For example, the mean void size approximately doubles when moving from a 90 % pearlitic environment to a 70 % environment. Moreover, the strain dependence becomes more pronounced where ferrite predominates. Given that nucleation is, at most, only weakly dependent on environment class, observed differences across pearlite fractions must arise predominantly from variations in growth behaviour. Thus, these results suggest that growth is governed by the local microstructural environments, with faster growth occurring where ferrite predominates and slower growth where pearlite content is high.

Our analysis likely underestimates the full growth-suppressing effect of highly pearlitic environments because it is based on two-dimensional (2D) micrographs and cannot capture the three-dimensional (3D) phase connectivity. As such, subsurface ferritic ligaments not intersected by the section plane may contribute to increasing apparent void growth even where the 2D section appears pearlitic. The high likelihood of this phenomenon is further supported by the frequent occurrence of ferritic environments given in Table 3.

Damage in MnS in multiphase environments. Combining the in-situ observations and statistical analysis with the phase-specific mechanical response from spherical indentation, we propose the following mechanism for damage in MnS when the tensile axis is parallel to the inclusion axis.

Rock-salt crystals such as MnS exhibit negligible tensile plasticity prior to fracture at room temperature (as shown for MgO, CaO [45, 46]). Fracture, thus, occurs once the local tensile stress reaches the fracture strength at or shortly after the elastic limit. In the initial elastic regime, the inclusion-matrix system can be approximated as iso-strain (Voigt bound) along the loading direction. However, because the elastic modulus of MnS is approximately half that of ferrite and pearlite (Table 4), the average elastic stress in MnS remains below its fracture strength at the matrix elastic limit. Fracture of MnS is therefore not expected at this stage. Once the phases surrounding MnS yield, local stress concentrations, amplified at intersections with slip bands observed in Figure 4, quickly increase the tensile stress in MnS toward its fracture strength, triggering crack initiation.

This mechanistic picture explains why damage nucleation shows little dependence on environment class: ferrite and pearlite have comparable indentation-equivalent yield strengths (Table 4), so the onset of plasticity is controlled more by crystallographic orientation and local morphology than by phase identity alone. By contrast, subsequent damage growth depends strongly on the surrounding phase. Because pearlite exhibits higher work-hardening than ferrite (Figure 9), post-yield macroscopic strain partitions preferentially into ferrite, leading to greater separation of MnS fragments following fracture, and larger void areas in ferritic environments Figure 8 d. Mixed environments show intermediate growth consistent with their local ferrite fraction. We therefore conclude that nucleation is dominated by local morphological features and slip-band interactions, whereas growth is governed by strain partitioning arising from the mechanical contrast

between the surrounding phases.

Consequently, while the identity of neighbouring phases has little influence on nucleation, ferritic local environments around MnS inclusions increase overall damage prevalence by accelerating post-nucleation growth. To elucidate 3D environmental effects, serial sectioning [47] or tomography experiments [48] could provide valuable additional insights. Combined with grain orientation mapping, such data could clarify how internal grain boundaries, misorientations between inclusions and matrix, and surface topography influence stress concentration and crack nucleation in MnS.

5. Conclusion

In this study, we quantified how phase distribution and local mechanical properties in 16MnCrS5 influence the prevalence and evolution of MnS-induced damage by combining machine-learning image segmentation with spherical nanoindentation. Our results demonstrate that neighbouring phases set the local strain partitioning around MnS inclusions, thereby affecting damage prevalence during deformation. The main conclusions are:

- Damage nucleation is largely insensitive to whether the local environment is ferritic or pearlitic within the examined strain range, consistent with the comparable indentation-equivalent yield strengths of both phases.
- Once nucleated, damage grows fastest in ferritic environments and is strongly inhibited in highly pearlitic surroundings due to pearlite's higher strain-hardening.
- Mixed environments exhibit intermediate growth proportional to their local ferrite fraction.

6. Acknowledgements

The authors express their gratitude to the Deutsche Forschungsgemeinschaft (DFG) for financial support in context of the Collaborative Research Centre CRC/Transregio 188/2 "Damage Controlled Forming Processes", project B02 and project T02, project no. 278868966. Funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) under the National Research Data Infrastructure – NFDI 38/1 – project number 460247524.

The data used in this publication was managed using the research data management platform Coscine (<http://doi.org/10.17616/R31NJNZ>) with storage space of the Research Data Storage (RDS) (DFG: INST222/1261-1) and DataStorage.nrw (DFG: INST222/1530-1) granted by the DFG and Ministry of Culture and Science of the State of North Rhine-Westphalia. This article is partly based upon work from COST Action CA21121 MecaNano, supported by COST (European Cooperation in Science and Technology—www.cost.eu)

7. Data and Code Availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contribution Statement

Maximilian A. Wollenweber: Methodology, Formal analysis, Investigation, Writing - Original Draft **Tom Reclik:** Software, Methodology, Formal analysis, Investigation, Writing - Original Draft **Clara Reinhart:** Software, Formal analysis, Investigation, Writing - Review & Editing **Verena Maier-Kiener:** Investigation, Writing - Review & Editing **Talal Al-Samman:** Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition **Ulrich Kerzel:** Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition **Sandra Korte-Kerzel:** Conceptualisation, Writing - Review & Editing, Supervision, Funding Acquisition

References

- [1] A. E. Tekkaya, N. B. Khalifa, O. Hering, R. Meya, S. Myslicki, F. Walther, Forming-induced damage and its effects on product properties, *CIRP Annals* 66 (1) (2017) 281–284.
- [2] O. Hering, A. E. Tekkaya, Damage-induced performance variations of cold forged parts, *Journal of Materials Processing Technology* 279 (2020) 116556.
- [3] R. Meya, C. Löbbe, A. E. Tekkaya, Stress state control by a novel bending process and its effect on damage and product performance, *Journal of Manufacturing Science and Engineering* 141 (10) (2019) 101013.
- [4] M. Azuma, S. Goutianos, N. Hansen, G. Winther, X. Huang, Effect of hardness of martensite and ferrite on void formation in dual phase steel, *Materials Science and Technology* 28 (9-10) (2012) 1092–1100.
- [5] F. Hannard, T. Pardoen, É. Maire, C. Le Bourlot, R. Mokso, A. Simar, Characterization and micromechanical modelling of microstructural heterogeneity effects on ductile fracture of 6xxx aluminium alloys, *Acta Materialia* 103 (2016) 558–572.
- [6] M. Zubair, S. Sandlöbes-Haut, M. A. Wollenweber, K. Bugelnig, C. F. Kusche, G. Requena, S. Korte-Kerzel, Strain heterogeneity and micro-damage nucleation under tensile stresses in an mg–5al–3ca alloy with an intermetallic skeleton, *Materials Science and Engineering: A* 767 (2019) 138414.
- [7] F. Pütz, F. Shen, M. Könnemann, S. Münstermann, The differences of damage initiation and accumulation of dp steels: a numerical and experimental analysis, *International Journal of Fracture* 226 (2020) 1–15.
- [8] C. Tian, C. F. Kusche, A. Medina, S. Lee, M. A. Wollenweber, R. Pippan, S. Korte-Kerzel, C. Kirchlechner, Understanding the damage initiation and growth mechanisms of two dp800 dual phase grades, *Materials & Design* 238 (2024) 112630.
- [9] C. F. Kusche, F. Pütz, S. Münstermann, T. Al-Samman, S. Korte-Kerzel, On the effect of strain and triaxiality on void evolution in a heterogeneous microstructure—a statistical and single void study of damage in dp800 steel, *Materials Science and Engineering: A* 799 (2021) 140332.
- [10] N. Habibi, N. Vajragupta, S. Münstermann, Deformation and damage assessments of two dp1000 steels using a micromechanical modelling method, *Crystals* 11 (7) (2021) 805.
- [11] S. Medghalchi, M. Zubair, E. Karimi, S. Sandlöbes-Haut, U. Kerzel, S. Korte-Kerzel, Determination of the rate dependence of damage formation in metallic-intermetallic mg–al–ca composites at elevated temperature using panoramic image analysis, *Advanced Engineering Materials* 25 (21) (2023) 2300956.
- [12] H. Ghadbeigi, C. Pinna, S. Celotto, Failure mechanisms in dp600 steel: Initiation, evolution and fracture, *Materials Science and Engineering: A* 588 (2013) 420–431.
- [13] C. C. Tasan, J. P. Hoefnagels, M. Diehl, D. Yan, F. Roters, D. Raabe, Strain localization and damage in dual phase steels investigated by coupled in-situ deformation experiments and crystal plasticity simulations, *International Journal of Plasticity* 63 (2014) 198–210.
- [14] Y. Liu, X. Zheng, S. Osovski, A. Srivastava, On the micromechanism of inclusion driven ductile fracture and its implications on fracture toughness, *Journal of the Mechanics and Physics of Solids* 130 (2019) 21–34.
- [15] J. Jiang, J. Yang, T. Zhang, J. Zou, Y. Wang, F. Dunne, T. Britton, Microstructurally sensitive crack nucleation around inclusions in powder metallurgy nickel-based superalloys, *Acta Materialia* 117 (2016) 333–344.
- [16] H. Toda, H. Ogo, K. Horikawa, K. Uesugi, A. Takeuchi, Y. Suzuki, M. Nakazawa, Y. Aoki, M. Kobayashi, The true ori-

- gin of ductile fracture in aluminum alloys, *Metallurgical and Materials Transactions A* 45 (2014) 765–776.
- [17] T. Gladman, Precipitation hardening in metals, *Materials science and technology* 15 (1) (1999) 30–36.
- [18] L. Holappa, O. Wijk, Inclusion engineering, *Treatise on process metallurgy* (2024) 303–321.
- [19] M. A. Wollenweber, C. F. Kusche, T. Al-Samman, S. Korte-Kerzel, On the automated characterisation of inclusion-induced damage in 16mncrs5 case-hardening steel, *Advances in Industrial and Manufacturing Engineering* 7 (2023) 100123.
- [20] C. F. Kusche, J. S.-L. Gibson, M. A. Wollenweber, S. Korte-Kerzel, On the mechanical properties and deformation mechanisms of manganese sulphide inclusions, *Materials & Design* 193 (2020) 108801.
- [21] O. Hering, A. Dunlap, A. E. Tekkaya, A. Aretz, A. Schwedt, Characterization of damage in forward rod extruded parts, *International Journal of Material Forming* 13 (2020) 1003–1014.
- [22] K. Moehring, F. Walther, Load direction-dependent influence of forming-induced initial damage on the fatigue performance of 16mncrs5 steel, *Materials* 13 (12) (2020) 2680.
- [23] M. A. Wollenweber, J. Werner, C. F. Kusche, C. Tian, P.-L. Sun, J. Gerlach, T. Al-Samman, S. Korte-Kerzel, On the plasticity of mns at elevated temperatures and its influence on damage prevalence, *Acta Materialia* 291 (2025) 120987.
- [24] N. Ånmark, A. Karasev, P. G. Jönsson, The effect of different non-metallic inclusions on the machinability of steels, *Materials* 8 (2) (2015) 751–783.
- [25] Y. Song, H. Zhang, L. Ren, A review of research on mns inclusions in high-quality steel, *Engineering Reports* 6 (5) (2024) e12892.
- [26] F. Qayyum, M. Umar, V. Elagin, M. Kirschner, F. Hoffmann, S. Guk, U. Prahl, Influence of non-metallic inclusions on local deformation and damage behavior of modified 16mncrs5 steel, *Crystals* 12 (2) (2022) 281.
- [27] F. Qayyum, S.-C. Tseng, S. Guk, F. Hoffmann, C.-K. Chao, U. Prahl, Examining the effect of mns particles on the local deformation behavior of 8mncrs4-4-13 steel by in situ tensile testing and digital image correlation, *Journal of Composites Science* 7 (7) (2023) 294.
- [28] F. Isensee, P. F. Jäger, S. A. Kohl, J. Petersen, K. H. Maier-Hein, Automated design of deep learning methods for biomedical image segmentation, *arXiv preprint arXiv:1904.08128* (2019).
- [29] C. Kusche, T. Reclik, M. Freund, T. Al-Samman, U. Kerzel, S. Korte-Kerzel, Large-area, high-resolution characterisation and classification of damage mechanisms in dual-phase steel using deep learning, *PloS one* 14 (5) (2019) e0216493.
- [30] S. Medghalchi, C. F. Kusche, E. Karimi, U. Kerzel, S. Korte-Kerzel, Damage analysis in dual-phase steel using deep learning: transfer from uniaxial to biaxial straining conditions by image data augmentation, *Jom* 72 (2020) 4420–4430.
- [31] S. Medghalchi, J. Kortmann, S.-H. Lee, E. Karimi, U. Kerzel, S. Korte-Kerzel, Automated segmentation of large image datasets using artificial intelligence for microstructure characterisation and damage analysis, *Materials & Design* 243 (2024) 113031.
- [32] T. Reclik, S. Medghalchi, P. Schumacher, M. Wollenweber, T. Al-Samman, S. Korte-Kerzel, U. Kerzel, Resolution enhancement of scanning electron micrographs using artificial intelligence, *Materials & Design* 253 (2025) 113955.
- [33] X. Li, S. Wang, J. Lohmar, G. Hirt, Design of caliber rolls incorporating load path dependent damage evolution, *Procedia manufacturing* 47 (2020) 643–648.
- [34] C. a. Corporation, *Computer Vision Annotation Tool (CVAT)* (Jul. 2024). doi:10.5281/zenodo.12771595. URL <https://zenodo.org/records/12771595>
- [35] A. Leitner, V. Maier-Kiener, D. Kiener, Essential refinements of spherical nanoindentation protocols for the reliable determination of mechanical flow curves, *Materials & Design* 146 (2018) 69–80.
- [36] L. R. Dice, Measures of the amount of ecologic association between species, *Ecology* 26 (3) (1945) 297–302.
- [37] J. Bertels, T. Eelbode, M. Berman, D. Vandermeulen, F. Maes, R. Bisschops, M. B. Blaschko, Optimizing the dice score and jaccard index for medical image segmentation: Theory and practice, in: *Medical Image Computing and Computer Assisted Intervention–MICCAI 2019: 22nd International Conference, Shenzhen, China, October 13–17, 2019, Proceedings, Part II* 22, Springer, 2019, pp. 92–100.
- [38] R. Usamentiaga, D. G. Lema, O. D. Pedrayes, D. F. Garcia, Automated surface defect detection in metals: a comparative review of object detection and semantic segmentation using deep learning, *IEEE Transactions on Industry Applications* 58 (3) (2022) 4203–4213.
- [39] T. Bayes, Lii. an essay towards solving a problem in the doctrine of chances. by the late rev. mr. bayes, frs communicated by mr. price, in a letter to john canton, amfr s, *Philosophical transactions of the Royal Society of London* (1763) 370–418.
- [40] S. Münstermann, Y. Feng, W. Bleck, Influencing parameters on elastic modulus of steels, *Canadian Metallurgical Quarterly* 53 (3) (2014) 264–273.
- [41] Z. Chen, U. Gandhi, J. Lee, R. Wagoner, Variation and consistency of young’s modulus in steel, *Journal of Materials Processing Technology* 227 (2016) 227–243.
- [42] A. Marder, B. Bramfitt, The effect of morphology on the strength of pearlite, *Metallurgical Transactions A* 7 (3) (1976) 365–372.
- [43] G. Avramovic-Cingara, Y. Ososkov, M. Jain, D. Wilkinson, Effect of martensite distribution on damage behaviour in dp600 dual phase steels, *Materials Science and Engineering: A* 516 (1–2) (2009) 7–16.
- [44] L. A. Lingnau, J. Heermant, J. L. Otto, K. Donnerbauer, L. M. Sauer, L. Lückner, M. Macias Barrientos, F. Walther, Separation of damage mechanisms in full forward rod extruded case-hardening steel 16mncrs5 using 3d image segmentation, *Materials* 17 (12) (2024) 3023.
- [45] R. Ku, T. Johnston, Fracture strength of mgo bicrystals, *Philosophical Magazine* 9 (98) (1964) 231–247.
- [46] R. W. Rice, Cao: Ii, properties, *Journal of the American Ceramic Society* 52 (8) (1969) 428–436.
- [47] S. Medghalchi, E. Karimi, S.-H. Lee, B. Berkels, U. Kerzel, S. Korte-Kerzel, Three-dimensional characterisation of deformation-induced damage in dual phase steel using deep learning, *Materials & Design* 232 (2023) 112108.
- [48] A. Shibata, A. Nakamura, T. Moronaga, K. Okada, T. Hara, R. Uejii, Three-dimensional morphologies, substructures, and crystallography of pearlite in carbon steel, *Science and Technology of Advanced Materials* (2025) 2523242.

Appendix A. Supplementary Materials

Appendix A.1. Additional details on image segmentation

Due to the class imbalance between damage and phase data, we implemented separate nnU-Net models: one for phase segmentation and one for damage segmentation. This approach allowed us to annotate additional damage-only training data for the damage network without requiring corresponding phase labels, which would be necessary in a single-network framework. Since phase information is relevant for damage prediction but not vice versa, we leveraged transfer learning by pre-training the damage network on phase data.

To address performance issues with damage outliers, we hypothesized that the small proportion of damage pixels relative to the input window might cause difficulties. We tested two modifications: (1) reducing the input window size from 3072×3072 pixels, and (2) adding phase segmentation masks inferred from the first trained network as additional input channels. Contrary to our hypothesis, the full 3072×3072 window without additional phase information yielded the best Dice scores (see Figure A.1). The optimal performance at full window size aligns with recommendations in the original nnU-Net publication [28]. Surprisingly, incorporating phase information did not improve performance. We attribute this to two factors: (1) potential error propagation from the phase network, since damage predominantly occurs in MnS phases, and (2) the fundamental limitation of relatively few damage-positive pixels, which likely requires a larger training dataset rather than architectural modifications to resolve effectively.

We analysed patches with low Dice scores to identify systematic failure modes (see Figure A.2). The outliers fell into three categories. Firstly, thin damage sites (patches a, b, c, and partially d): The network struggled to segment damage features with small spatial extent. Secondly, large damage sites with brightness variation (patches d, e, f): In these cases, fractured MnS islands exhibited different brightness levels across the two separated segments, potentially hindering the segmentation. Lastly, false negative in ground truth: One damage site was correctly identified by the network but was absent from the ground truth annotations, patch g.

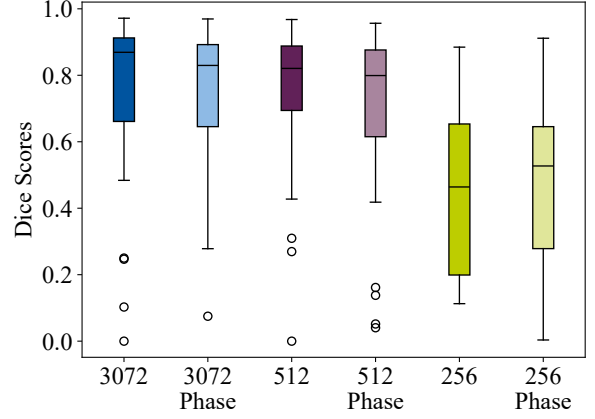


Figure A.1: Dice scores of damage-net trained with different window sizes and additional phase information.

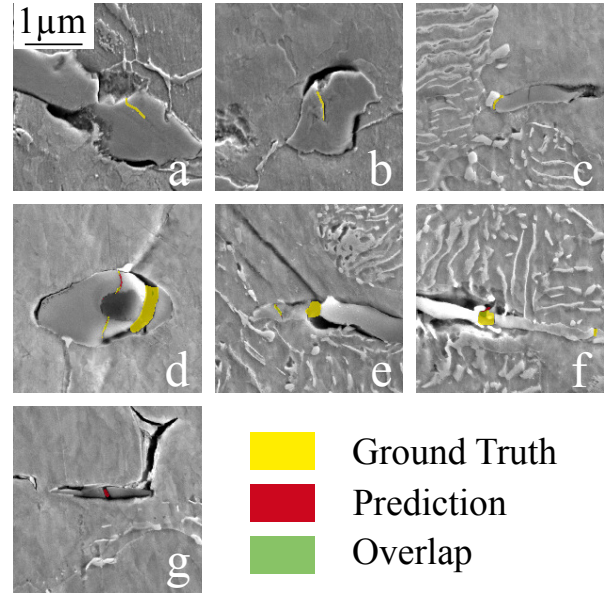


Figure A.2: Dice score outliers of damage-net trained with the full window size and no additional phase information.