

Title:

Effect of martensite hardness on mechanical properties and stress/strain-partitioning behavior in ferrite + martensite dual-phase steels

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Abstract

Low-carbon dual-phase (DP) steels composed of ferrite and martensite are widely used in automotive industries due to their good combination of strength and ductility and remarkable strain-hardening ability. Such exceptional mechanical properties of DP steels arise from microstructural deformation heterogeneity due to the different deformation roles of soft ferrite and hard martensite. The present study investigated mechanical properties and local deformation behavior of DP steels having different martensite hardness (strength) using digital image correlation (DIC) technique and *in-situ* X-ray diffraction (XRD) experiment during tensile deformation. The martensite hardness was reduced by tempering treatment, and its mechanical properties were compared with the as-quenched DP specimen by tensile testing. The tempered DP specimen exhibited reduced strength but enhanced ductility, especially in post-uniform elongation, compared to the as-quenched DP specimen. DIC-strain analysis indicated that the tempering effectively inhibited strain localization, allowing a greater contribution of martensite to plastic deformation. On the other hand, the *in-situ* XRD analysis revealed that the tempering significantly restricted the stress-increasing potential of martensite. Our results suggest that martensite, acting as a hard domain, plays a significant role in enhancing strain-hardening ability and achieving large uniform elongation due to its high stress-increasing potential. However, the presence of hard martensite also imposes restrictions on post-uniform elongation due to the strong strain localization induced in microstructures.

1. Introduction

Low-carbon dual-phase (DP) steels composed of a soft ferrite phase and a hard martensite phase are widely used in many industrial applications due to their good strength-ductility balance and excellent formability at room temperature. Over several decades, numerous studies have investigated DP steels, aiming to further enhance mechanical properties by controlling microstructural factors such as grain size, morphology, volume fraction and strength (hardness) of each phase [1-7]. Grain refinement of DP steels is an effective method for achieving high strength without significantly sacrificing ductility [3, 7]. Martensite distribution is another critical microstructural factor affecting the mechanical properties of DP steels. A DP structure, having chain-shaped martensite surrounding ferrite grains, exhibited enhanced ductility compared to a DP structure with isolated, bulky martensite [4]. Additionally, the effect of phase fraction (f) or phase hardness (strength (σ)) on mechanical properties can be roughly predicted using the conventional rule-of-mixture, expressed as $\sigma = f_F \sigma_F + f_M \sigma_M$, where subscripts **F** and **M** represent ferrite and martensite, respectively. Despite the extensive investigation into the relationship between individual microstructural factors and mechanical behavior, a unified understanding that integrates all microstructural factors has yet to be fully established. Quantitative evaluation of deformation heterogeneity at a microstructure level is a primary approach to be investigated, and the occurrence of deformation heterogeneity in DP structure would be mainly due to the difference in hardness (strength) between the soft ferrite and hard martensite phases. Therefore, the characterization of local deformation behavior in the change of phase hardness can be a first step in exploring the origin of the heterogeneous deformation nature of DP steels.

The digital image correlation (DIC) technique, which can provide a full-field local strain distribution at a microstructure scale, is one powerful method for characterizing deformation heterogeneity in DP steels [8-11]. The *In-situ* X-ray diffraction (XRD)/neutron diffraction experiment during deformation, which can evaluate the internal stress of each constituent phase, is another useful technique for investigating stress-partitioning behavior between two phases [12-16]. These two advanced measurement techniques are believed to have great synergy in the quantitative evaluation of the deformation heterogeneity of DP steels.

In the present study, we investigated the effect of martensite hardness on mechanical properties and local deformation behavior using the DIC technique and *in-situ* high-energy XRD diffraction analysis during deformation with a focus on the microstructural deformation heterogeneity of DP steels.

2. Experimental procedure

Low-carbon steel having a chemical composition of Fe-2Mn-0.1C (Mn: 2.04, C: 0.096, Al: 0.019, Fe: balanced; mass%) was used in the present study. DP structure composed of ferrite and martensite was fabricated through multi-step thermo-mechanical processing, as shown in **Fig. 1**. Initially, homogenization at 1200°C for 48 hr followed by furnace cooling was conducted under a vacuum atmosphere to inhibit segregation of alloying elements. Subsequently, cold-rolling with a 90% reduction in thickness was carried out. The cold-rolled specimen was then austenitized at 830°C for 3 hr and subsequently furnace-cooled to obtain a ferrite + pearlite (F+P) structure, which was the base structure to fabricate a DP structure having chain-shaped martensite. Next, DP structure was obtained by ferrite + austenite two-phase intercritical annealing at 750°C for 1 hr followed by water-quenching (WQ), during which the austenite transformed to martensite. The obtained DP specimen will be referred to as “as-quenched DP”. In parallel with fabricating the as-quenched DP, the as-quenched DP specimen was subjected to additional heat treatment: tempering at 400 °C for 1 hr followed by WQ. This tempering process aims to reduce the strength (hardness) of martensite. The specimen obtained from this process will be referred to as “tempered DP” hereafter.

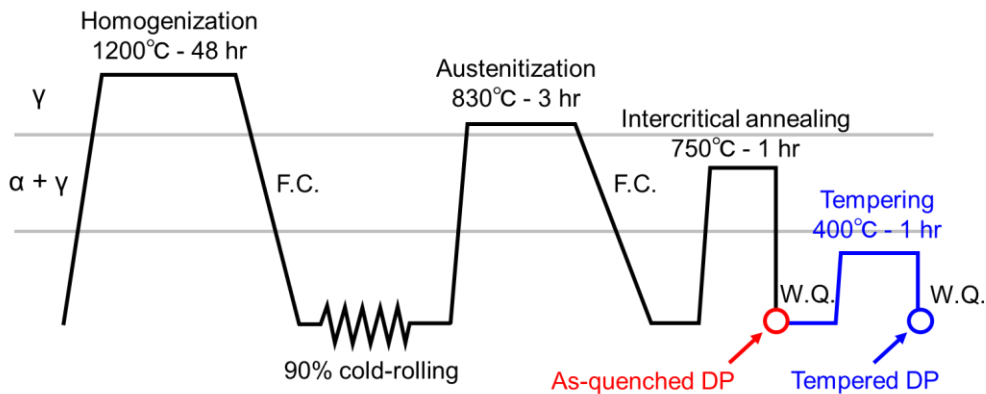


Fig. 1 Thermo-mechanical process for fabricating ferrite + martensite dual phase (DP) with different martensite hardness (strength). To reduce martensite hardness, the as-quenched DP specimen was subjected to tempering treatment at 400°C for 1 hr, followed by water-quenching.

Microstructure observation was carried out using a field emission type scanning electron microscope (FE-SEM) (JEOL, JSM-7800F) with an accelerated voltage of 15 kV. For SEM observation, the specimen was mechanically polished with 400-4000 grit-sized emery papers and then electrolytically polished in a chemical solution of 90 vol% of CH₃COOH and 10

vol. % of HClO_4 with a voltage of 22 V for 30 s at room temperature. The grain size was measured using the linear interception method on SEM images, and the martensite volume fraction was estimated by the point-counting method. The strength (hardness) of each phase was assessed using a nano-indentation testing machine (TI 950 TriboIndenter, Hysitron) with an indent load of 1000 μN and a dwell time of 0.5 s.

Global mechanical properties of DP specimens were evaluated by a uniaxial tensile testing machine (Shimadzu, AG-100kN Xplus) at room temperature with an initial strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$. The tensile direction was aligned parallel to the rolling direction of the sheet, and the tensile specimen with a gage length of 10 mm and a cross-section of 3 mm (width) $\times$ 1 mm (thickness) was prepared. Tensile elongation and local strain distribution were precisely measured by the digital image correlation (DIC) technique with a dedicated DIC strain-visualizing software (Correlation Solutions, Vic-2D).

Before tensile testing, the specimen surface was painted with black and white inks to provide a speckle pattern for DIC strain analysis. During tensile testing, 2432×2054 pixel-sized images including speckle-patterned specimen were captured by a CCD camera (SVS625MFCP, SVS-VISTEK) at a constant capture speed of 5 fps (frame per second) until tensile fracture. All captured images were provided for DIC analysis to obtain global tensile elongation and local strain distribution maps. In DIC strain analysis, the subset size and strain measurement step were set to $0.11 \text{ mm} \times 0.11 \text{ mm}$ and 0.017 mm, respectively. Microscopic DIC (μ -DIC) analysis was also carried out. The surface of the tensile specimen was polished, and identical area observation was carried out in SEM at specific tensile strains. SEM contrast of microstructure served as the base pattern for DIC analysis. In addition, very fine random dot-patterns of colloidal silica (SiO_2) with an average particle radius of 0.04 μm were introduced on the polished surface to achieve high-resolution μ -DIC strain analysis. An example of colloidal silica-patterned DP microstructure is shown in **Fig. 2**. In the μ -DIC analysis, the subset size and strain measurement step were set to $2.3 \mu\text{m} \times 2.3 \mu\text{m}$ and 0.39 μm , respectively, for both specimens.

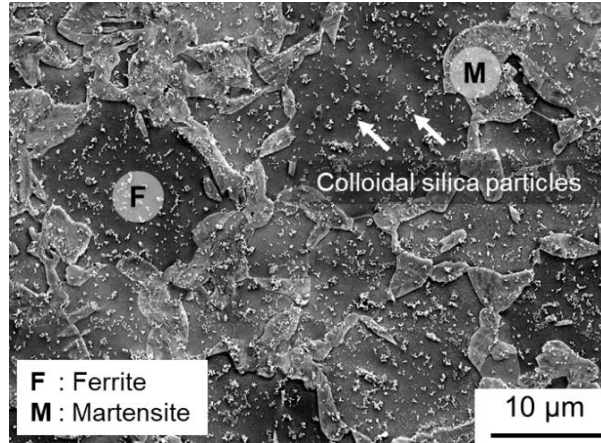


Fig. 2 An example of SEM micrograph showing precise random-dot patterns with colloidal silica (SiO_2) particles on DP structure for achieving high-resolution micro-DIC analysis. Colloidal silica is expressed by white arrows, and **F** and **M** denote ferrite and martensite, respectively.

In-situ synchrotron high-energy XRD (HE-XRD) measurement was performed to investigate the internal stress evolution of each phase during tensile deformation. The massive-scale facility of high energy synchrotron beamline (BL 46 XU) at Spring-8 (Super Photon ring-8 GeV) of Japan Synchrotron Radiation Research Institute (JASRI) was utilized under proposal No. 2021B1853. The facility provided diffraction profiles with a high time resolution of 1 second through a monochromatic synchrotron beam with strong intensity of 30 keV ($\lambda = 0.0413$ nm). The equipment layout for *in-situ* synchrotron HE-XRD measurement during tensile deformation is schematically illustrated in **Fig. 3**. Tensile specimen with a gage length of 10 mm, a gage width of 3 mm and a thickness of 0.5 mm was used for the *in-situ* HE-XRD experiment. The incident beam was positioned at the gage center of the tensile specimen, and the diffracted X-rays from the tensile specimen were collected by serially-connected 6 detectors (MYTHEN X, Dectris).

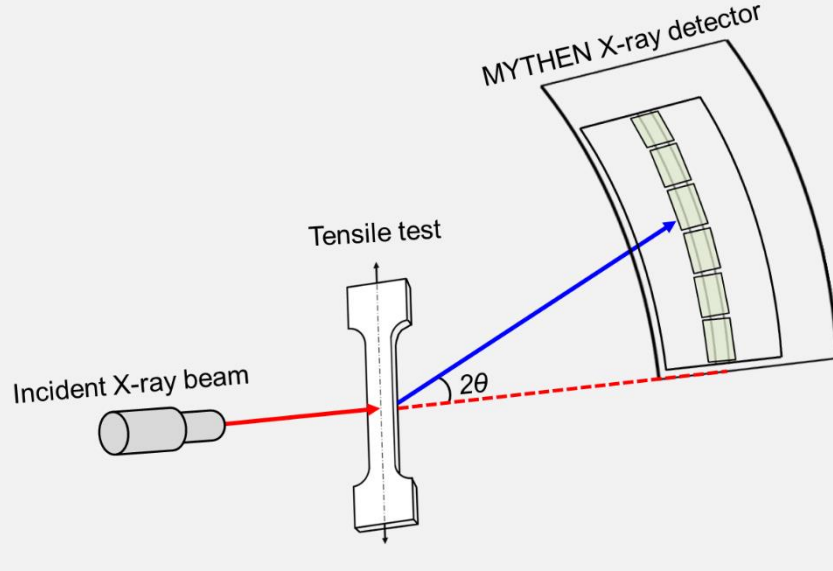
SPring-8*In-situ* synchrotron XRD measurement with tensile test

Fig. 3 Schematic illustration of *in-situ* synchrotron high-energy X-ray diffraction (HE-XRD) measurement during tensile test at SPring-8 (Super Photon ring-8 GeV) of JASRI (Japan Synchrotron Radiation Research Institute) located in Hyogo Prefecture, Japan.

The method for estimating internal stress from XRD experiments is illustrated in **Fig. 4**. During tensile testing, the lattice elastically deforms, resulting in an increase in the lattice constant (from d_0 to d) along the tensile axis (**Fig. 4 (a)**). Consequently, the (hkl) XRD peaks, perpendicular to the tensile axis, shift to smaller 2θ values (from $2\theta_0$ to 2θ) (**Fig. 4 (b)**). According to Bragg's law ($\lambda = 2d\sin\theta$, where λ is the wavelength), the elastic lattice strain (e), determined as $(d-d_0)/d_0$, can be expressed as $e = (\sin\theta_0/\sin\theta) - 1$ (**Fig. 4 (c)**). The internal stress (σ) was estimated using Hooke's law ($\sigma = Ee$, where E is the elastic modulus). **Fig. 4 (d)** illustrates typical peak profiles in low-carbon DP structures before and during deformation, along with the separation of ferrite and martensite peaks for estimating internal stresses in each phase. This method was employed in the present study to characterize internal stress evolution in each phase and quantify their respective contributions during deformation. As shown in **Fig. 4 (d)**, in low-carbon steels with carbon content less than 0.4 wt.%, both ferrite and martensite have BCC crystal structures with similar d-spacing [17, 18]. This similarity results in overlapped peak profiles when observed in XRD analysis. Peak separation was carried out based on two key assumptions: Firstly, the martensite, containing more carbons and higher

dislocation density, exhibits slightly larger d-spacing (resulting in smaller 2θ) with peak broadening. Secondly, the integrated intensity of the separated peak reflects the volume fraction of each phase. (110) peak, exhibiting the highest peak intensity and the smallest 2θ (i.e., the (110) planes are nearly perpendicular to the tensile axis with a tilting angle of $\sim 5.85^\circ$), was selected for the calculation of elastic strain. The elastic strain was calculated by analyzing the peak shift of the (110) plane for each phase, and the internal stress was estimated using Hooke's law with a (110)-corresponding elastic modulus of 232 GPa for Fe [19].

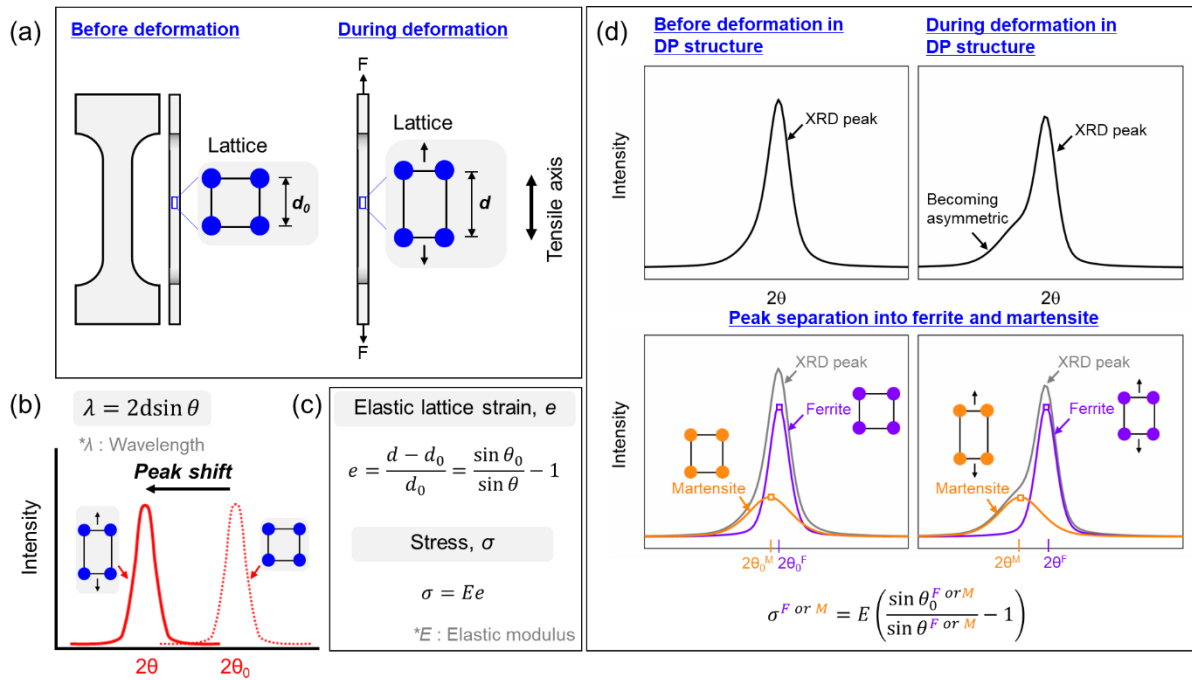


Fig. 4 Illustration of the internal stress-estimating method from the *in-situ* XRD experiment during deformation. (a) Schematic illustration of the tensile specimen and the lattice changes before and during tensile deformation. (b) Illustration of peak profile changes observed during deformation. (c) Calculation of elastic lattice strain based on the peak shift. Stress is estimated using Hooke's law. (d) Peak profiles in DP structures before and during deformation, along with peak separation into ferrite and martensite, for estimating internal stresses in each phase.

3. Results

3.1. Microstructural characterization of as-quenched DP and tempered DP specimens

Fig. 5 shows SEM microstructures of (a) as-quenched DP and (b) tempered DP specimens at both low and high magnifications. Capital letters **F** and **M** labeled on the images indicate ferrite and martensite, respectively. Low-magnification images (top row) revealed that both DP specimens showed similar microstructural features in martensite fraction ($\sim 34\%$), ferrite grain size ($\sim 15 \mu\text{m}$) and martensite distributions with chain-shaped morphologies, as detailed

in **Table 1**. In high-magnification images (bottom row), a significant number of fine precipitates (white contrast) were observed within martensite of the tempered DP specimen, which were likely carbides formed during tempering treatment [20-24]. These carbide precipitates are believed to influence the mechanical properties of martensite.

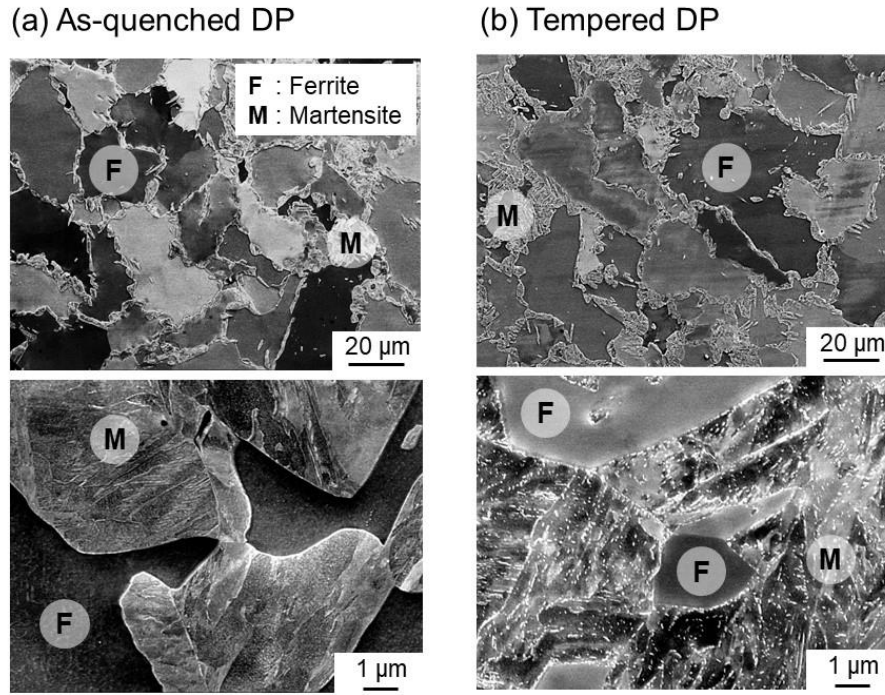


Fig. 5 SEM microstructures of (a) as-quenched DP specimen and (b) tempered DP specimen at low magnification (top row) and high magnification (bottom row).

Table 1 Summary of ferrite grain size, martensite fraction and martensite distributions.

	Ferrite grain size / μm	Martensite fraction (%)	Martensite distribution
As-quenched DP	14.6	35.3	Chain-shaped
Tempered DP	16.8	32.2	Chain-shaped

Fig. 6 displays nano-hardness of ferrite and martensite. A decrease in the hardness of martensite was observed ($3.56 \text{ GPa} \rightarrow 2.95 \text{ GPa}$) in the tempered DP specimen, while the ferrite did not exhibit a dramatic hardness change ($1.89 \text{ GPa} \rightarrow 1.75 \text{ GPa}$) upon tempering. This suggests that tempering primarily affects the softening of martensite, probably due to a decrease in dislocation density and a weakened solid solution strengthening effect by carbon atoms. In contrast, ferrite, with its inherently low dislocation density and limited carbon contents even in the as-quenched state, experiences a less softening effect.

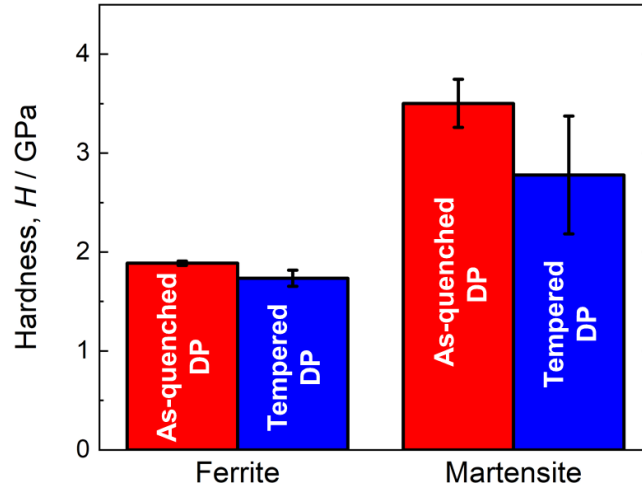


Fig. 6 Nano-hardness of ferrite and martensite in as-quenched DP specimen (red) and tempered DP specimen (blue).

3.2. Tensile properties

Fig. 7 (a) shows nominal stress-strain (S-S) curves of the as-quenched DP specimen and the tempered DP specimen obtained from uniaxial tensile testing at room temperature. The open circle symbol on each S-S curve indicates the uniform elongation point. The tempered DP specimen exhibited lower strength and larger tensile ductility than the as-quenched DP specimen. Discontinuous yielding at a stress of 450 MPa was observed in the tempered DP specimen, and this would be due to reduced dislocation density and the occurrence of dislocation immobilization pinned by carbon atoms during tempering treatment [25-27]. Just after discontinuous yielding, the strain-localized band (Lüders band) and its one-time propagation were observed in the DIC-strain distribution map (see **Fig. S1** in supplementary materials). After that, the specimen was strain-hardened to 621 MPa at the tensile strain of 8.7% (uniform elongation), then deformed locally until 22.9% total elongation. In contrast, the as-quenched DP specimen exhibited a continuous yielding and high strain-hardening ability, with a relatively low yield strength of 400 MPa but a high ultimate tensile strength (UTS) of 796 MPa. Its uniform elongation and total elongation were 9.2% and 12.5%, respectively. Notably, the as-quenched DP specimen exhibits slightly larger uniform elongation rather than the tempered DP specimen.

The strain-hardening behaviors of both DP specimens were investigated to understand their uniform elongations. According to Considère criterion [28], macroscopic necking (i.e., onset of plastic instability) occurs at the crossing point of the strain-hardening rate (defined as $d\sigma/d\varepsilon$)

curve and true stress-true strain (σ - ϵ) curve. **Fig. 7 (b)** displays strain-hardening rate curves of both DP specimens, together with true stress-true strain curves. The as-quenched DP specimen always exhibited a larger strain-hardening rate throughout deformation, thereby meeting its true S-S curve at a larger strain. This would be the reason for the achievement of sufficient uniform elongation in the as-quenched specimen.

Local deformation behavior, including necking evolution, was also investigated. **Fig. 7 (c)** presents DIC-strain distribution maps of DP specimens tensile-deformed to 5.0%, 9.0%, 12.5% and 23.0%. The tensile axis is parallel to the vertical direction in the images, and the colors indicate the value of the normal strain component in the tensile direction (ϵ_{II}) according to a key color bar. At the nominal strains of 5% and 9%, both DP specimens showed a mostly homogeneous deformation in the gage part. At the global tensile strain of 12.5%, strain localization (i.e., macroscopic necking) appeared in both DP specimens but with significantly different magnitudes. Very strong strain localization with a maximum local strain value larger than 0.45 was observed in the as-quenched DP specimen, which was almost twice the maximum strain value in the tempered DP specimen (~ 0.2). Just after 12.5% global tensile strain, the as-quenched DP specimen underwent tensile fracture. The tempered DP specimen continued to deform, showing a gradual necking development, and the local strain eventually reached ~ 0.6 at the global tensile strain of 23.0%. The DIC results suggest that the significant post-uniform elongation in the tempered DP specimen is attributed to gradual strain localization and a high strain-enduring capacity.

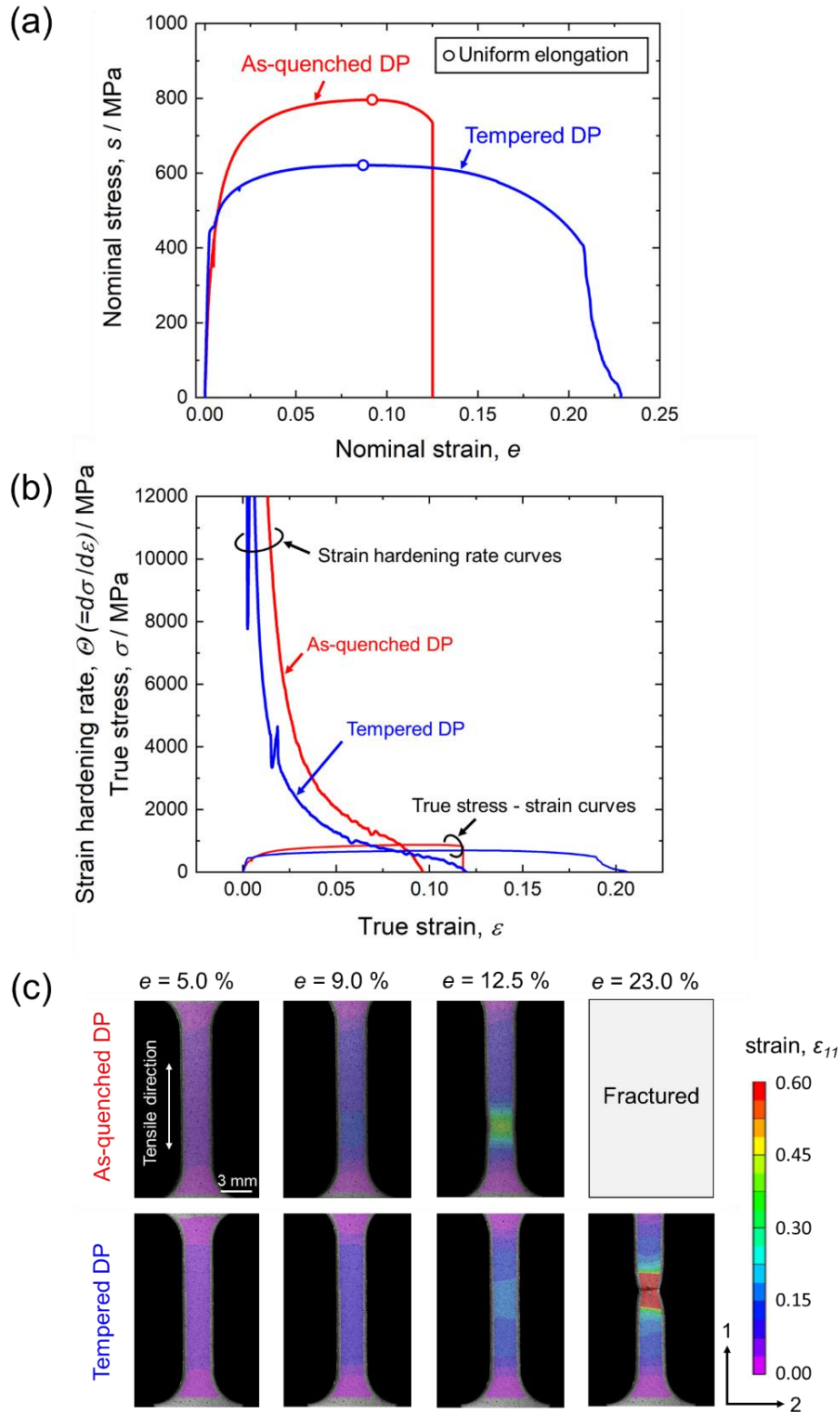


Fig. 7 (a) Nominal stress-strain (S-S) curves and (b) strain-hardening rate curves + true S-S curves of as-quenched DP and tempered DP specimens. (c) DIC-local strain distribution maps of tensile specimens at various tensile strains. Colors on strain maps indicate the value of a normal strain component in a tensile direction (ε_{11}) according to the key strain bar.

3.3. Microstructural strain distribution characterized by μ -DIC analysis

To explore the reasons for the significant change in global mechanical behavior, microstructural deformation behavior was carefully investigated using μ -DIC strain analysis. **Fig. 8** shows SEM microstructures, boundary maps and corresponding ε_{II} strain distribution maps of 1.3% tensile-deformed (a) as-quenched DP specimen and (b) tempered DP specimen. Note that fine particles seen in SEM images are the colloidal silica (SiO_2) markers used for precise DIC analysis. **F** and **M** in SEM microstructures indicate ferrite and martensite, respectively. Blue and red lines in the boundary map denote the ferrite/martensite (F/M) boundary and ferrite/ferrite (F/F) boundary, respectively. Almost all the boundaries in both specimens were F/M boundaries, with a small portion of F/F boundaries. For both DP specimens, despite the early stage of deformation ($e \sim 1.3\%$), the strain distribution was highly heterogeneous. In the as-quenched DP specimen, there was pronounced strain-partitioning between ferrite and martensite. The martensite exhibited extremely low strain values (deep-purple color), with sharp deformation occurring only in regions near the F/M boundary. Almost all strain localization occurred at the ferrite grains and was distributed along the F/M boundary. In contrast, the tempered DP specimen showed somewhat weak strain-partitioning. Some martensite regions, indicated by arrows, contributed to plastic deformation. No sharp strain gradient across F/M boundaries was observed.

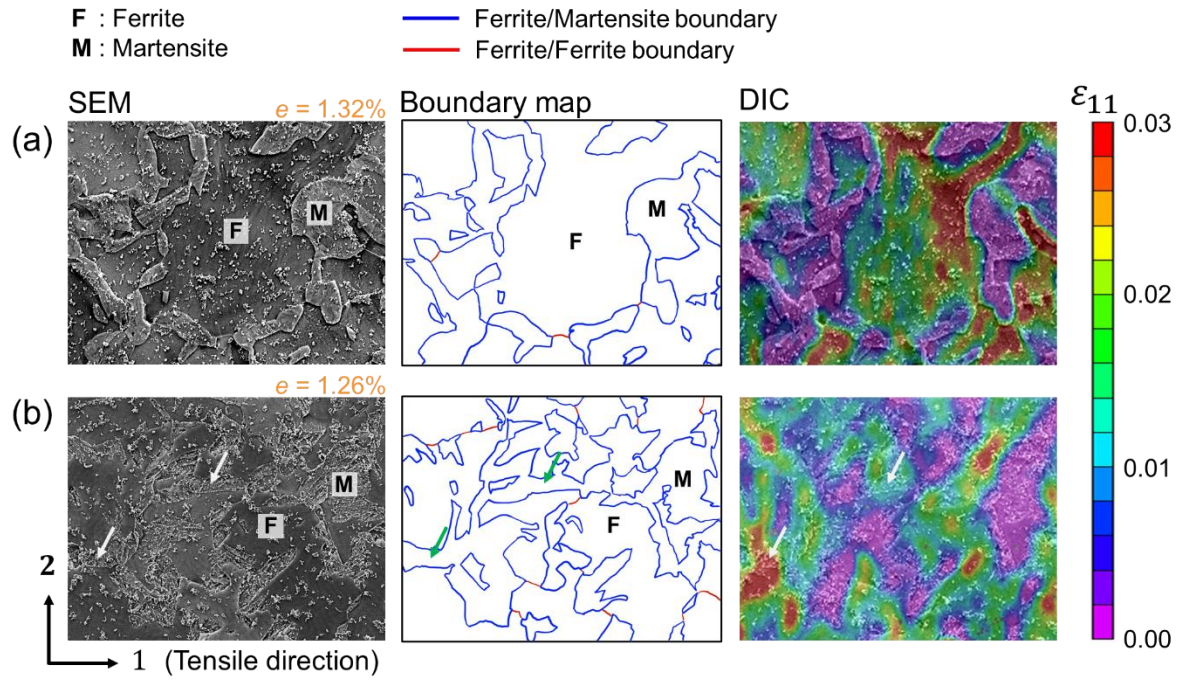


Fig. 8 SEM microstructures, boundary maps and corresponding DIC-strain distribution maps of $\sim 1.3\%$ tensile-deformed (a) as-quenched DP and (b) tempered DP specimens obtained from the high-resolution μ -DIC analysis. **F** and **M** are ferrite and martensite, respectively, and blue and red lines in boundary maps indicate the ferrite/martensite boundary and the ferrite/ferrite boundary, respectively. The colors in strain distribution maps refer to the value of normal strain in the tensile direction. Arrows on the strain map of the tempered DP specimen indicate the representative martensite particles exhibiting large local strains.

Further, wider-area strain analysis was carried out, as displayed in **Fig. 9**. The top row shows DIC-strain maps including the distribution of F/M boundaries (depicted by white lines), and the middle 2nd and 3rd rows exhibit selected strain maps for ferrite and martensite, respectively. The bottom row provides a quantitative assessment in the form of local strain histograms for each phase. In the as-quenched DP specimen, strain localization appeared to be frequently obstructed by martensite particles. In contrast, in the tempered DP specimen, strain localization is sometimes extended into martensite particles, as indicated by arrows. Another crucial point to note is the strain distribution in martensite. While most martensite in the as-quenched DP specimen exhibited small strain values (expressed in purple), martensite in the tempered DP specimen displayed a broader spectrum of strain values. The significantly different deformation heterogeneity between the two specimens can also be clearly recognized in the local strain histograms. Movies displaying the evolution of strain distribution to the global tensile strain of $\sim 9\%$ can be found in the supplementary materials. The strain evolution

of each phase during tensile deformation was quantitatively investigated (**Fig. 10**). Each data point represents an average value of local strains for ferrite or martensite (i.e., phase strain), and the broken line is the one-to-one correspondence line between phase strain and average strain in DIC-analyzed region, indicating the ideal homogeneous deformation. It was confirmed in both DP specimens that ferrite always had a larger strain value than martensite throughout deformation, and the strain difference between ferrite and martensite increased with plastic deformation. All phases showed a linear increase in phase strain. The slopes for ferrite and martensite in the tempered DP specimen showed a smaller deviation from the ideal homogeneous deformation line, indicating more homogeneous deformation throughout tensile deformation. Small strain-partitioning between two phases in the tempered DP specimen is believed to be one reason for achieving large global ductility, as it suppresses strain localization.

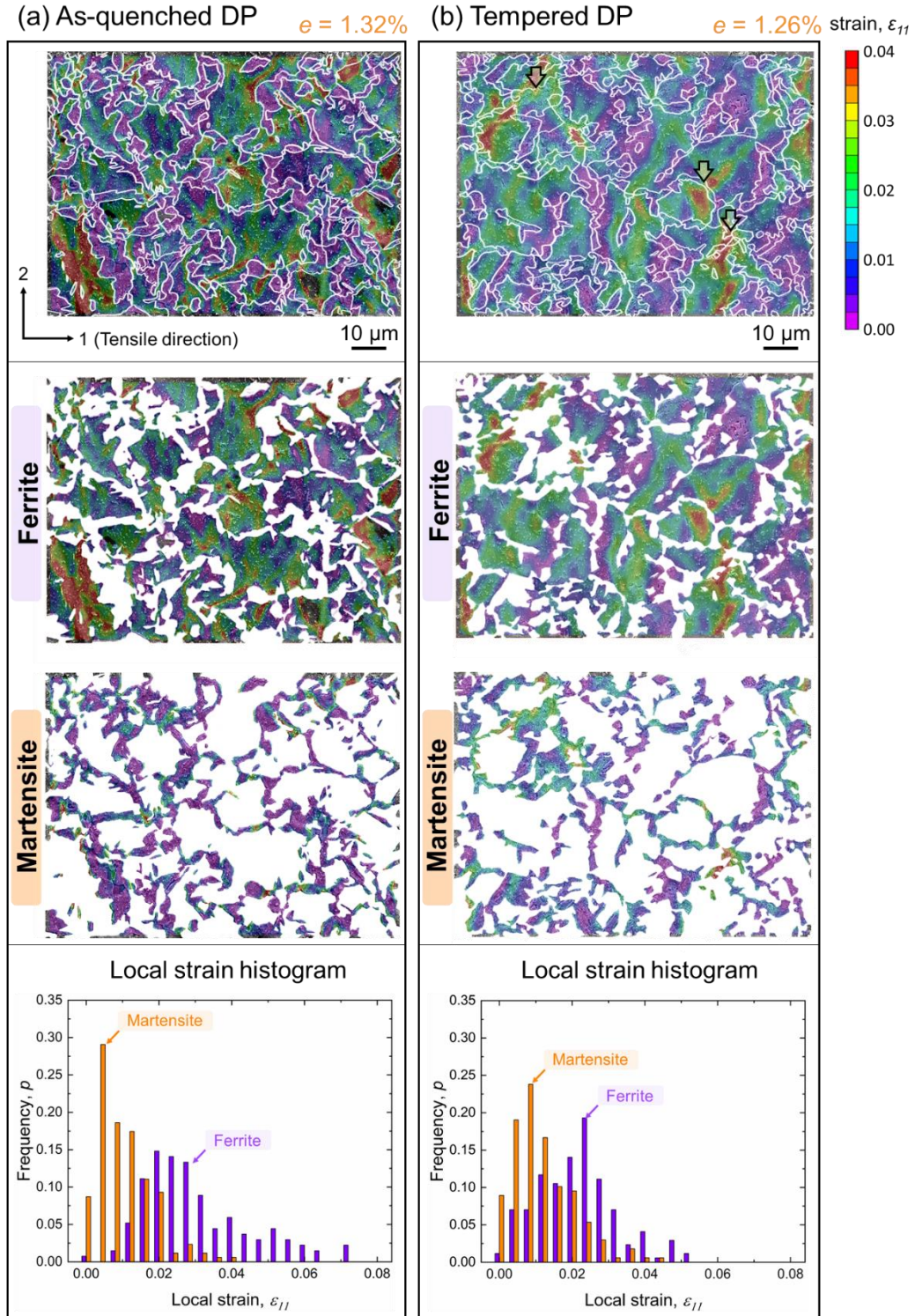


Fig. 9 Wide-range DIC-strain distribution maps of $\sim 1.3\%$ tensile-deformed (a) as-quenched DP and (b) tempered DP specimens. The white line on the maps indicates ferrite/martensite (F/M) boundaries. The middle 2nd and 3rd rows display strain maps extracted for ferrite and martensite, respectively. The bottom row shows local strain histograms for each phase.

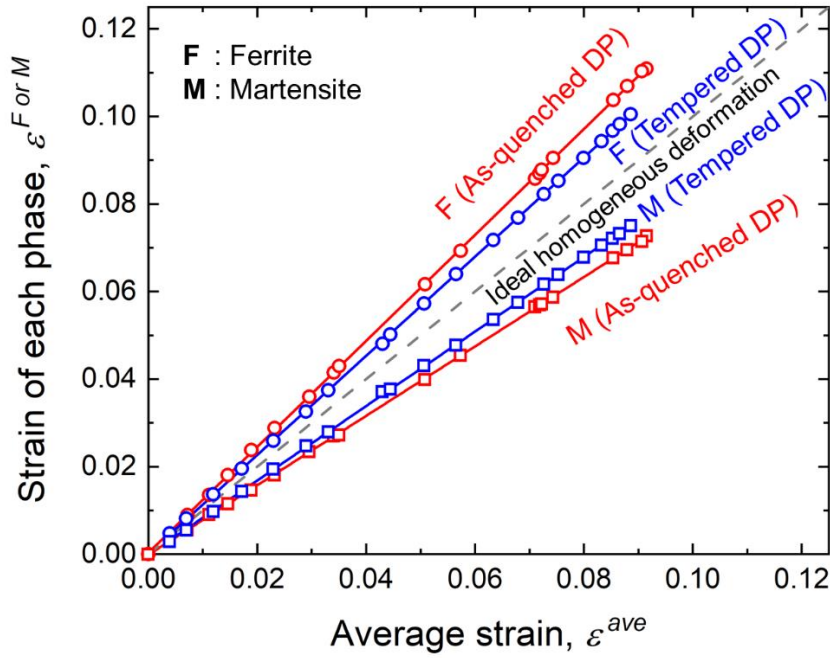


Fig. 10 Local strain evolution of ferrite (F) phase and martensite (M) phase with tensile deformation in as-quenched DP (red) and tempered DP (blue) specimens. A one-to-one correspondence line indicating ideal homogeneous deformation is included in the graph.

3.4. Stress-partitioning between ferrite and martensite

The DIC-local strain results exhibited a distinct difference in strain distribution between the two DP specimens, which can be attributed to relatively different hardness ratios of ferrite and martensite due to tempering. However, it is important to note that the nano-hardness values of each phase shown in **Fig. 6** are obtained from a deformation-free state. To gain a deeper understanding of the evolution of internal stress in ferrite and martensite during tensile deformation, we employed *in-situ* HE-XRD measurements, as presented in **Fig. 11** (a). For reference, the evolution of peak profiles at different applied stresses is indicated in **Fig. S2** in the supplementary materials. The internal stress data for each phase were plotted as a function of global tensile stress. An iso-stress line was indicated by a dashed line on the graphs to explore stress-partitioning between ferrite and martensite. The yield strength (σ_{YS}) and ultimate tensile strength (σ_{UTS}) were indicated by vertical straight lines on the graph. In the as-quenched DP specimen, remarkable stress-partitioning between ferrite and martensite occurred even before yielding, with internal stresses of 314 MPa for ferrite and 664 MPa for martensite at the global tensile stress of 387 MPa. After yielding, the stress-partitioning became more significant due to a dramatic increase in martensite phase stress. At the global tensile stress of

766 MPa (near σ_{UTS}), the internal stress of ferrite reached 497 MPa, and that of martensite reached 1311 MPa in the as-quenched DP specimen. Such an apparent stress-partitioning behavior observed in the current study coincides well with other previous results achieved by several research groups [13-15]. In contrast, the tempered DP specimen showed somewhat unique stress-partitioning behaviors. No significant stress-partitioning was observed during elastic deformation, with only a small stress-partitioning of 79 MPa just before yielding (426 MPa). More interestingly, a stress drop was observed in the ferrite just after yielding from 457 MPa down to 394 MPa. The stress-dropped ferrite rapidly recovered up to 471 MPa at a global tensile stress of 560 MPa, and then kept almost constant values until near σ_{UTS} . Martensite in the tempered DP specimen exhibited a monotonous increase in internal stress, but with limited stress-increasing potential reaching a maximum of 875 MPa. The obtained results exhibit that the tempered DP specimen has two distinctive features in internal stress evolution: The occurrence of stress drop & recovery in ferrite and limited stress-increasing capacity in martensite. Although the exact reason for the occurrence of stress drop in ferrite is still unclear, it may be related to the discontinuous yielding phenomenon and Lüders deformation, which will be discussed in the discussion section. In addition, it is also interesting that each ferrite in both DP specimens eventually reaches a similar maximum internal stress value (476 MPa for tempered DP ~ 496 MPa for as-quenched DP), which could be considered a stress limit of ferrite in the present alloying system. The limited stress-increasing capacity of martensite in the tempered DP specimen can be understood through distinct martensite-softening by tempering, probably due to significantly reduced lattice defects hindering dislocation movement.

Fig. 11 (b) shows the stress contribution of each phase, defined as a multiplication of the internal stress of each phase and its volume fraction ($(1 - f_M) \times \sigma^F$ or $f_M \times \sigma^M$ where f_M is martensite fraction). The true stress-strain curve was included in the respective graph to compare global stress levels with contributed internal stresses. The total value of contributed internal stresses of both ferrite and martensite roughly matched its global true stresses. In the as-quenched DP specimen, the martensite exhibited a much greater contribution to the global stress than the ferrite. In contrast, the martensite in the tempered DP specimen contributed less to global stress. It is interesting to note that the difference in initial martensite hardness (ΔH_M) between both specimens is just 0.61 GPa. However, such a small change in martensite hardness greatly influences the contribution of martensite to global stress, as well as changing its absolute value of internal stress. This strongly suggests that the tensile properties of DP

structure, having a complex microstructure composed of ferrite and martensite, cannot be anticipated only with initial hardness information. Instead, it is essential to take into account the mechanical interactions that occur complexly during deformation.

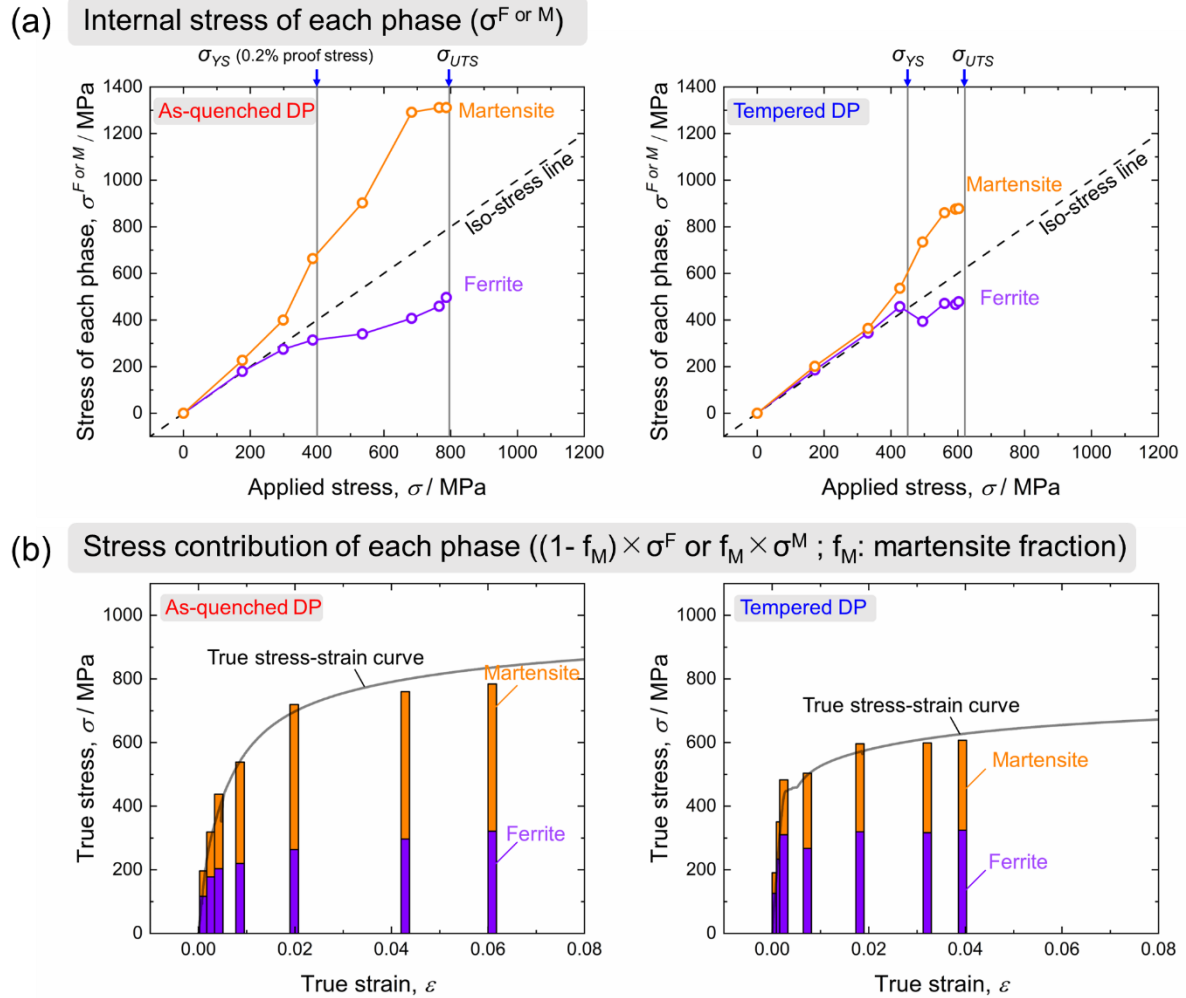


Fig. 11 (a) Internal stress evolution of ferrite (F) and martensite (M) with tensile deformation obtained from *in-situ* high-energy XRD measurement. The iso-stress line is included on the graph to evaluate stress-partitioning between ferrite and martensite. Two vertical straight lines on the respective graph correspond to yield strength (σ_{YS}) and ultimate tensile strength (σ_{UTS}). (b) Stress contributions of ferrite and martensite, defined as a fraction (f) $\times$ internal stress (σ) for each constituent phase. The true stress-strain curve was included in the respective graph, aiming to compare global stress levels with contributed internal stresses.

4. Discussion

4.1. Lüders deformation in DP steels induced by tempering

According to the continuum mechanics-based stress analysis achieved by Schwab et al. [29, 30], the stress condition dynamically changes in Lüders band sweeping, and stress-relaxation

occurs at the front part of Lüders band in the low-carbon ferritic steels. Zhang et al. [31] experimentally confirmed the apparent stress-relaxation of the soft domain (austenite) during Lüders deformation of the medium-Mn transformation-induced plasticity (TRIP) steels using the *in-situ* XRD elastic strain mapping experiment. While this is not the exact case of Lüders deformation in DP steels, stress-relaxation of austenite is believed to share similarities in terms of its deformation role as a soft phase. One potential explanation is that carbon atoms diffusing during tempering could pin dislocations in the soft ferrite [32, 33], thereby making plastic deformation more difficult. This pinning effect might contribute to the temporary stress drop and recovery in ferrite, and could also play a role in the occurrence of Lüders deformation during tensile testing. Martensite, which forms from austenite, inherently contains a high concentration of carbon atoms. However, a significant portion of these supersaturated carbons is expected to precipitate during tempering, as shown in **Fig. 5**. A recent study on tempering effect in Fe-1.7Mn-0.17C DP steels [34] indicates that carbon atoms primarily form precipitates rather than segregate to dislocations. Consequently, tempered ferrite may be the primary contributor to the yield phenomena, potentially explaining its observed stress drop and recovery, although the exact mechanism remains unclear.

4.2. Martensite-softening effect by tempering

High strength nature of martensite might be mainly due to solid-solution strengthening by supersaturated carbons and dislocation strengthening, as well as grain refinement strengthening resulting from the formation of multiple variants from a single austenite grain. As seen in **Fig. 5**, the tempered DP specimen possesses a number of carbides in the martensite interior, which suggests reduction of solid-solution strengthening by carbon. Such pronounced carbide formation in tempered martensite has been confirmed by several previous studies [23, 24, 26], with one study specifically noting a decrease in hardness. Dislocation density for ferrite and martensite was measured through Williamson-Hall method with obtained XRD data, as shown in **Table 2**. After tempering, a significant decrease in dislocation density of martensite was observed, whereas the no significant changes in that of ferrite. Therefore, the formation of numerous carbides and significantly reduced dislocation density would be main contributor to decreasing strength of martensite.

Table 2 Dislocation density of ferrite and martensite in the as-quenched and tempered DP specimens.

	Dislocation density	
	Ferrite, ρ_F / m^{-2}	Martensite, ρ_M / m^{-2}
As-quenched DP	1.4×10^{15}	2.3×10^{16}
Tempered DP	1.9×10^{15}	9.1×10^{15}

4.3. Effect of martensite hardness on local strain/stress and mechanical properties

The present study quantitatively characterized strain- and stress-partitioning behaviors between ferrite and martensite using μ -DIC technique and *in-situ* XRD analysis. In the as-quenched DP specimen, martensite exhibited a minor contribution to strain, but was responsible for high internal stress. In contrast, in the tempered DP specimen, martensite exhibited more deformation but contributed less to internal stress. Further softening of martensite through higher-temperature tempering for extended durations is expected to result in more homogeneous deformation, with minimal stress-partitioning, similar to that of a single-phase ferritic structure. This suggest that strength differences between the two phases govern deformation heterogeneity at the microstructure scale. **Fig. 8** and **Fig. 9** demonstrate that strain localizes primarily in the ferrite regions near ferrite/martensite boundaries, rather than in the centers of ferrite grains. This severe strain localization in ferrite at the boundaries can be understood as a result of compensating for the strains that are not accommodated by the neighboring martensite.

While reducing the strength of martensite can seemingly improve overall ductility, it is important to note that the tempered DP specimen exhibits reduced uniform elongation. As shown in **Fig. 7** (b), this reduction is due to the decreased strain-hardening ability resulting from tempering. A more detailed consideration can be drawn from the stress contribution results (**Fig. 11** (b)), where martensite in the tempered DP specimen demonstrates a significantly reduced stress increase during deformation, reflecting its limited strain-hardening capacity. Thus, the presence of hard martensite is crucial, not only for achieving high strength but also for enhancing uniform elongation due to its high stress-increasing ability. This discussion highlights that the strain and stress in each phase are not independently related to the strength and ductility properties, but are intricately interconnected, influencing each other in complex ways. Nevertheless, the findings clarify the importance of hard phase and its significant role in deformation during tensile testing, offering valuable insights for material

design strategies aimed at improving both strength and ductility.

Several previous studies have examined the effect of martensite hardness, focusing on the temperature dependence of tensile properties in DP structures [1, 26], strain hardening in martensite [20], carbide formation [20-24], and microstructural evolution of martensite [21, 26]. Building on these foundational findings, our careful systematic analysis of strain- and stress-partitioning reveals new insights, specifically highlighting the distinct deformation roles of ferrite and martensite as martensite strength changes.

The analytical combination of both advanced strain- and stress-measuring techniques, such as DIC strain analysis and *in-situ* XRD measurement, can also be applied for the investigations of other microstructure factor-dependence, such as grain size, phase fraction and phase distribution, on the mechanical properties. These investigations are essential for developing a comprehensive understanding of the underlying deformation mechanisms in DP structures.

5. Conclusions

In this study, we examined the tensile properties of DP structures with different martensite hardness and characterized strain- and stress-partitioning behaviors between ferrite and martensite using micro-DIC strain analysis and *in-situ* XRD measurement during tensile deformation. Our main findings can be summarized as follows:

- (1) We successfully fabricated a DP structure with chain-shaped martensite morphology and a ferrite grain size of $\sim 15\ \mu\text{m}$ through heat treatment at 750°C for 60 min, followed by water quenching. Subsequent tempering treatment at 400°C for 60 min resulted in a reduction in the hardness of martensite from 3.56 GPa to 2.95 GPa, with a small change in the hardness of ferrite from 1.89 GPa to 1.75 GPa.
- (2) In tensile testing, the tempered DP specimen showed relatively low tensile strength and large total elongation, compared to the as-quenched DP specimen. We observed discontinuous yielding and subsequent strain-localized band (Lüders band) in the tempered DP specimen, whereas the as-quenched specimen DP exhibited continuous yielding behavior. The enhanced total elongation shown in the tempered DP specimen was primarily attributed to significant post-uniform elongation resulting from a gradual strain localization with sufficient strain-enduring capacity. Uniform elongation slightly decreased by tempering, due to a significant decrease in strain-hardening ability.
- (3) μ -DIC local strain analysis revealed relatively homogeneous deformation at a microstructural scale in the tempered DP specimen, attributed to the greater contribution of martensite to plastic deformation. This contributed to the suppression of strain localization in microstructures, thereby achieving greater ductility (total elongation).
- (4) *In-situ* HE-XRD study indicated an unusual behavior in the internal stress evolution of the tempered DP specimen. Unlike the as-quenched DP specimen which exhibited remarkable stress-partitioning at the beginning of deformation before yielding, no distinct stress-partitioning was observed in the tempered DP specimen. Additionally, temporary stress drop and recovery of ferrite were observed in the tempered DP specimen just after yielding. The tempered DP specimen showed significantly lower internal stress levels in martensite, compared to its as-quenched counterpart. This suggests that tempering significantly limits the stress-increasing potential of martensite, resulting in limited strain-hardening ability and smaller uniform elongation in the tempered DP specimen.

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Author contributions

M-H.P. was responsible for this research. M-H.P. and Y.F. carried out most of the experiments and analysis. M-H.P. carried out *in-situ* XRD measurements during tensile deformation. M-H.P., A.S. and N.T. wrote the current paper. All authors discussed the results, and checked and revised the manuscript.

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