



Research Article

Dynamics of Portevin-Le Chatelier banding revealed through grain refinement in high-Mn austenitic steel: Sequential overcoming of necking

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ABSTRACT

Dynamics of Portevin-Le Chatelier (PLC) banding were thoroughly revealed through grain refinement in high-Mn austenitic steel. Fully recrystallized 22Mn-0.6C steels (wt%), with mean grain sizes ranging from 8.4 μm (coarse grain, CG) to 0.82 μm (ultrafine grain, UFG), were successfully fabricated through repeated cold rolling and subsequent annealing. Serrations were observed on the global stress-strain curves of all specimens, while the UFG specimen showed notably weak serrations in the early stages of deformation. Using the digital image correlation (DIC) technique, the serration behavior on the stress-strain curves of all specimens was found to perfectly correspond to the local heterogeneous deformation characterized by the formation, propagation, and annihilation of PLC bands. With grain refinement, localized deformation within the PLC band decreased, while the deformation beyond it became more pronounced, resulting in more homogeneous deformation across the entire gage part. This study presents the first experimental evaluation of the local strain-hardening rate through the simultaneous application of *in-situ* synchrotron XRD and DIC measurements during the tensile test. It was found that necking was periodically triggered but was soon overcome through the high strain hardening within the propagating PLC bands, which was achieved through the localized multiplication of dislocations and the formation of stacking faults. The UFG specimen exhibited greater local strain hardening than the CG specimen, induced by the formation of more dislocations and stacking faults. Such an increase in local strain hardening with grain refinement resulted in more homogeneous deformation behavior and consequently weaker serrations in the early stages of deformation.

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1. Introduction

High-Mn austenitic steels have been recognized as the most promising materials for structural applications in the automobile industry field, due to their high strain-hardening ability and out-

standing combination of strength and ductility. The high strain-hardening ability of high-Mn austenitic steels has been attributed to the formation of deformation twins during plastic deformation. The deformation twins subdivide grains and act as obstacles to mobile dislocations. As a result, grains are significantly hardened and plastic instability is postponed, leading to both high strength and large ductility [1–7]. Such a phenomenon is known as the twinning induced plasticity (TWIP) effect or the dynamic Hall-Petch effect. On the other hand, carbon-containing high-Mn austenitic steels frequently exhibit serration behavior on stress-

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strain curves [8–13]. Carbon atoms repeatedly pin mobile dislocation and exert a resisting force during dislocation glide, creating the so-called Cottrell atmosphere, and it appears on a macroscopic scale as serration peaks on stress-strain curves. Such dynamic interaction between dislocations and carbon atoms is known as Portevin–Le Chatelier (PLC) or dynamic strain aging (DSA) effect [14–16]. Besides high-Mn austenitic steels, some metallic materials with both high strength and large ductility, such as medium-Mn steels [17,18], Fe-Ni-C transformation induced plasticity (TRIP) steels [19,20], and high-entropy alloys [21–24], also exhibit serration behavior on their stress-strain curves, and the DSA effect has also been raised as another strengthening mechanism [25,26]. Research on serration behavior has primarily focused on parameters such as temperature [23,27–29], strain rate [13,27–30], and carbon content [31,32]. It has been found that sufficiently high temperatures and low strain rates, in the presence of carbon, promote serration behavior. This is because high temperatures facilitate carbon diffusion, while low strain rates decrease dislocation velocity, creating conditions that enable carbon atoms to effectively pin mobile dislocations. However, these changes in temperature and strain rate simultaneously reduce the effective stress required for dislocations to overcome short-range barriers, further complicating the discussion on the specific contribution of serration behavior (or the DSA effect) to mechanical properties. Another variable that can provide insights into the intrinsic role of serration behavior on mechanical properties without directly affecting the thermal activation is grain size. However, studies on the effect of grain size on serration behavior are limited, with only a few researchers investigating its influence on the onset strain, where serration behavior first appears on stress-strain curves [33,34]. According to Charnock [33], the onset strain of serration behavior in Cu-30wt% Zn alloys decreased as grain size was refined from 200 μm to 4 μm . The author attributed the early onset of serration behavior in fine-grained specimens to both a decrease in dislocation velocity and an increase in dislocation density, which facilitated the formation of the Cottrell atmosphere. On the other hand, McCormick reported that the onset strain in 6063 aluminum alloys with various mean grain sizes ranging from 650 μm to 46 μm was independent of grain size, and the author considered that grain size might have a minor effect on the dislocation density or velocity [34]. Research on the effect of grain size on serration behavior has been limited and even lacks consistency. This is probably because serration peaks at the very early stage of deformation are so small that the analyses based solely on stress-strain curves could lead to misinterpretation of the effect of grain size on serration behavior. Meanwhile, it has been well established that grain refinement imposes spatial constraints on dislocations within the grains. As a result, it becomes difficult for dislocations to pile up and activate dislocation sources in neighboring grains, leading to an increase in stress until plastic deformation occurs [35,36]. Grain refinement could also affect post-yield strain-hardening behavior through the reduced mean free path for dislocation motion and the resulting increase in dislocation density. Previous studies [33,34] have primarily focused on the increased dislocation density, which naturally increases the probability of carbon pinning and leads to the formation of Cottrell atmospheres around dislocations. However, our previous studies found that the significant increase in yield strength of high-Mn steels [37,38] was achieved through grain refinement down to the UFG region, e.g., the yield strength approximately doubled from 353 MPa to 698 MPa with grain refinement in 22Mn-0.6C-3Si steel. Such high flow stress should also be taken into account, as it can enable the de-pinning of carbon atoms and require a higher strain hardening rate to avoid necking. Given the current lack of sufficient and consistent findings, and considering that grain refinement can be a key variable for clarifying the intrinsic nature of serration behavior without altering thermal activation, it is essential to un-

derstand how grain size affects serration behavior and the corresponding local strain-hardening response. Recently, we successfully clarified the mesoscopic nature of serration behavior in high-Mn austenitic steel through the simultaneous application of the digital image correlation (DIC) technique and *in-situ* synchrotron XRD measurement during tensile test [8]. With the aid of the DIC technique, serration behavior on the stress-strain curves of high-Mn austenitic steel was found to perfectly correspond to the formation, propagation, and annihilation of strain-localization bands that are called PLC bands (PLC banding) during tensile tests. The DIC method can be applied to precisely analyze the effect of grain refinement on serration behavior associated with PLC banding. *In-situ* synchrotron XRD measurement is a powerful complementary method that can capture real-time changes in crystal defects such as dislocation density and stacking fault probability, as well as lattice strain, within an averaged scattering volume, and which enables the correlations between measured material parameters and local strain-hardening behavior of specimens with different grain sizes.

In the present study, we aim to clarify the effect of grain refinement on serration behavior and to further reveal the mechanism of serration behavior in high-Mn austenitic steels. Fe-22Mn-0.6C (wt%) steels having a wide range of mean grain sizes, from a coarse grain (CG) of 8.4 μm to an ultrafine grain (UFG) of 0.82 μm , were fabricated through repeated cold rolling and subsequent annealing process. The effect of grain refinement on serration behavior accompanying the propagation of PLC bands was systematically analyzed using the DIC technique and *in-situ* synchrotron XRD measurement during tensile tests. The dynamics of serration behavior were discussed in association with the evolution of deformation microstructure in specimens with various mean grain sizes and the consequent changes in local strain-hardening behavior.

2. Material and methods

2.1. Material

The 22Mn-0.6C steel (wt%) was used in the present study. The detailed chemical composition of the steel is given in Table 1. The hot-forged plate (in its as-received condition) underwent four cycles of the cold-rolling and annealing process that we previously developed [39]. The intermediate annealing between each cold-rolling step was conducted at 600 °C for 600 s using a salt bath, in order to preserve the material's deformability. In the final annealing stage, the specimens were annealed again in a salt bath at various temperature–time conditions ranging from 700 °C to 850 °C and from 15 s to 3600 s, respectively, followed by water quenching.

2.2. Microstructure observation

Microstructure observations were conducted after the final annealing treatments. The specimens were observed from the transverse direction (TD) of the rolled sheet at its mid-thickness. Prior to observation, mechanical polishing was performed using SiC papers with grit sizes ranging from #1000 to #4000, followed by electrolytic polishing in a solution composed of 10 % perchloric acid and 90 % ethanol at room temperature (27 °C). The microstructures were examined using a field emission scanning electron microscope (FE-SEM, JEOL JSM-7800F) equipped with a back-scattered electron (BSE) detector. The average grain sizes of the annealed specimens were evaluated by the line intercept method applied to SEM-BSE images, in which annealing twin boundaries were also counted as high-angle grain boundaries. To ensure consistent comparison of grain sizes across different annealing con-

Table 1
Chemical composition of the investigated high-Mn austenitic steel (wt%).

C	Si	Mn	P	S	Al	Cr	O	N	Fe
0.56	0.06	21.68	<0.004	0.007	<0.01	<0.01	<0.0011	0.0049	Bal.

ditions, the original SEM-BSE images were cropped and uniformly resized to standardize the scale bar length.

2.3. Tensile test

Sheet-type tensile specimens, with a gage length of 10 mm, a width of 5 mm, and a thickness of 1 mm, were cut from the rolled sheet using an electrical discharge machine. The tensile direction was parallel to the rolling direction (RD) of the sheet. Tensile tests were performed under quasi-static conditions at an initial strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$ at room temperature using a SHIMADZU AG-100 kN Xplus. Global tensile strain was precisely measured using the digital image correlation (DIC) technique. A non-contact digital extensometer, implemented in Vic-2D software (version 6, Correlated Solutions), was positioned along the gage part to continuously capture strain data during the tensile test. The global strain measured by DIC technique was synchronized with the load data obtained from the tensile testing machine, using the onset of tensile deformation as the reference point.

2.4. Digital image correlation (DIC) technique

The DIC technique was employed to evaluate local strain and strain rate distributions during the tensile tests of the specimens with various mean grain sizes. Initially, all specimens were painted white, followed by spraying with black speckle patterns using an airbrush to form a well-dispersed random pattern on the tensile specimens. By tracking the displacement of the speckle patterns during deformation, local strain and strain rate were measured. Throughout the tensile tests, the surface of each specimen was recorded at a rate of 5 frames per second using a complementary metal-oxide-semiconductor (CMOS) camera with a resolution of 2432×2054 pixels. The recorded images were analyzed using the Vic-2D software with a subset of 29 pixels in a square and a step size of 7 pixels, optimized for accurately evaluating local strain and strain rate at the given resolution of the CMOS camera.

2.5. Simultaneous application of in-situ synchrotron XRD measurement and DIC technique during tensile test

In-situ synchrotron XRD measurements were conducted to evaluate a real-time change of material parameters at a fixed beam position during the tensile tests of the specimens with different mean grain sizes. The experiment was performed at the high-energy synchrotron beamline of BL13XU at Super Photon ring-8 GeV (Spring-8) operated by Japan Synchrotron Radiation Research Institute (JASRI). Sheet-type tensile specimens with a gage length of 10 mm, a width of 3 mm, and a thickness of 0.5 mm were cut using an electrical discharge machine from sheets with fully recrystallized microstructures having different mean grain sizes. The tensile direction was aligned parallel to the rolling direction (RD) of the sheet. A monochromatic synchrotron beam with an energy of 30 keV ($\lambda = 0.0413 \text{ nm}$) was irradiated into an area 0.5 mm in height and 2 mm in width. During the tensile test, transmitted diffraction beams were collected in real-time by six detectors (MYTHEN X, Dectris) arranged in serial. The obtained diffraction profiles were fitted to a Voigt function using Origin Pro 2022 software, and the center positions and full width at half maximum

(FWHM) of the diffraction peaks were estimated. Based on the extracted peak information, the dislocation density, stacking fault probability, and lattice strain at the fixed beam position were evaluated. Details of the evaluation procedures are provided in the Supplementary Material. DIC analysis was performed simultaneously with *in-situ* synchrotron XRD measurement during the tensile test to correlate the PLC banding with material parameters obtained from the diffraction profiles. A CMOS camera was installed in the beamline for recording the displacements of speckle patterns on the specimen at a rate of 1 frame per second. Detailed experimental setups can be found in our previous studies [16,38].

3. Results and discussion

3.1. Microstructure before tensile test

Fig. 1(a)–(f) shows SEM-BSE images of 22Mn-0.6C steels with the various mean grain sizes ranging from $0.82 \mu\text{m}$ to $8.4 \mu\text{m}$, which were obtained by altering the final annealing conditions of the repeated cold rolling and annealing process. The specimens with mean grain sizes of $0.82 \mu\text{m}$ and $8.4 \mu\text{m}$ are hereafter re-

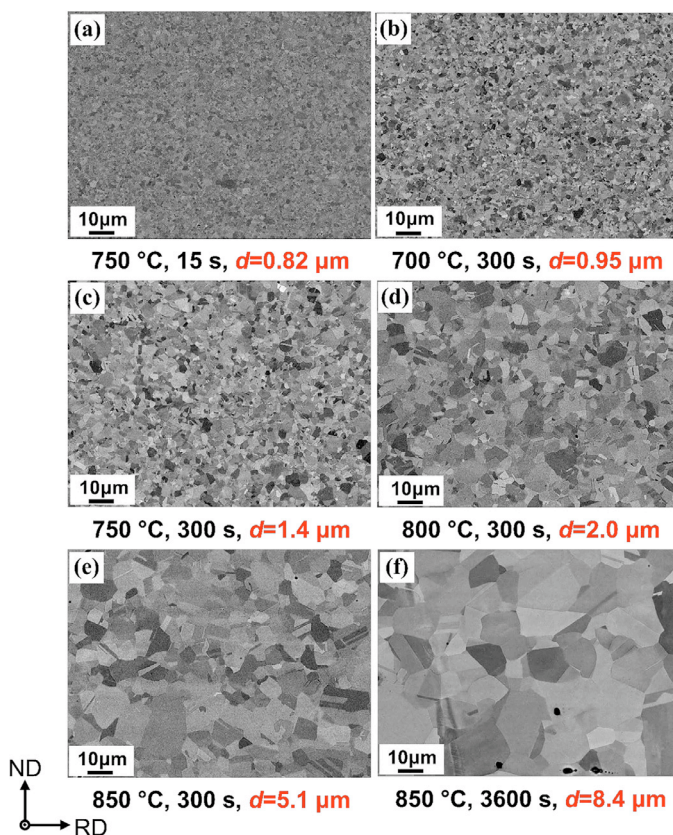


Fig. 1. SEM-BSE images of 22Mn-0.6C steels with various mean grain sizes: (a) $0.82 \mu\text{m}$, (b) $0.95 \mu\text{m}$, (c) $1.4 \mu\text{m}$, (d) $2.0 \mu\text{m}$, (e) $5.1 \mu\text{m}$, and (f) $8.4 \mu\text{m}$, observed from the transverse direction (TD) of the rolled sheet. Grain size was controlled in the final stage of the repeated cold rolling and annealing process. The annealing conditions and the corresponding mean grain sizes are indicated at the bottom of each image.

ferred to as ultrafine grained (UFG) and coarse grained (CG) specimens, respectively, and are set as the representative specimens to discuss the effect of grain size on serration behavior. The microstructures were observed from the transverse direction (TD) of the rolled sheet. All microstructures exhibited homogeneous and equiaxed grains containing a large number of annealing twins. The grain contrasts were uniform, indicating that the microstructures were fully recrystallized. Considerable grain refinement to the UFG region with fully recrystallized microstructures was successfully achieved, even with a short annealing time of 15 s, as a high density of nucleation sites for recrystallization was introduced after the repeated cold-rolling and annealing process.

3.2. Mechanical properties of specimens with the various mean grain sizes

Nominal stress-strain curves of specimens with the various mean grain sizes, ranging from $0.82\ \mu\text{m}$ to $8.4\ \mu\text{m}$, are shown in Fig. 2. It was clearly observed that both yield strength and tensile strength increased significantly with grain refinement from $8.4\ \mu\text{m}$ to $0.82\ \mu\text{m}$. The yield strength (0.2 % proof stress) increased from 294 MPa (CG) to 754 MPa (UFG), and the tensile strength increased from 1015 MPa (CG) to 1251 MPa (UFG). Total elongation decreased from 0.71 (CG) to 0.47 (UFG) with grain refinement. However, it is notable that the UFG specimen still retained a large total elongation. With grain refinement below $2.0\ \mu\text{m}$, a small yield drop accompanied by Lüders deformation was observed, becoming more pronounced in the UFG specimen with a mean grain size of $0.82\ \mu\text{m}$. It is also notable that post-uniform elongation was quite small in all specimens, indicating that they fractured soon after experiencing necking. The short post-uniform elongation will be discussed in a later section (Fig. 8). Serration behavior was observed on the stress-strain curves of all specimens. Interestingly, specimens with mean grain sizes of $0.82\ \mu\text{m}$ and $0.95\ \mu\text{m}$ exhibited weak serration behavior during their early stage of deformation ($e = 0.10$ to 0.20), whereas specimens with coarser grain sizes showed more pronounced serration behavior throughout the entire deformation. As discussed in Section 1 (Introduction), the DSA effect associated with serration behavior is more pronounced within specific ranges of strain rate and temperature, and is suppressed outside these ranges. However, to the best of the authors' knowledge, no study has reported the suppression of DSA (or serration behavior) in the UFG region, including in alloys such as high-Mn austenitic steels where DSA is typically prominent under quasi-static conditions at room temperature. This unique feature will be further explored in the following sections.

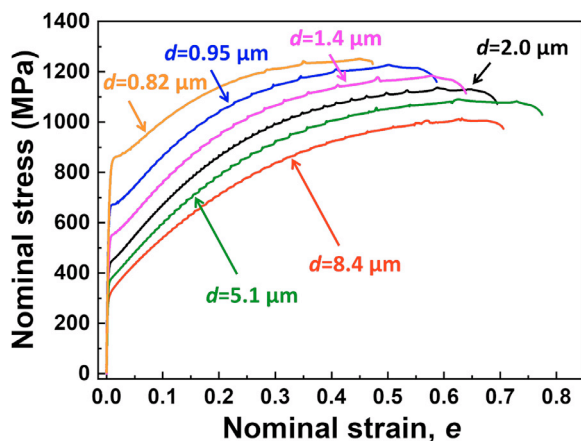


Fig. 2. Nominal stress-strain curves of 22Mn-0.6C steels with various mean grain sizes.

3.3. Correlation between serrations and localized deformation behavior in the CG and UFG specimens

To investigate the reason for the changes in serration behavior with grain refinement, the localized deformation behavior of the CG and UFG specimens was analyzed using the DIC method, as shown in Fig. 3(a) and (b), respectively. The 3D graphs in Fig. 3 are made up of strain rate profiles along the analysis line throughout the entire tensile deformation, with the inset of Fig. 3(a) showing how it was measured. These graphs provide an overview of the localized deformation behavior during tensile deformation. In DIC analysis, strain rate refers to the change in strain per second and can capture the deformation occurring at a specific time (or global nominal strain) during the tensile test. Meanwhile, strain represents the accumulated strain up to that point. Wall-like red

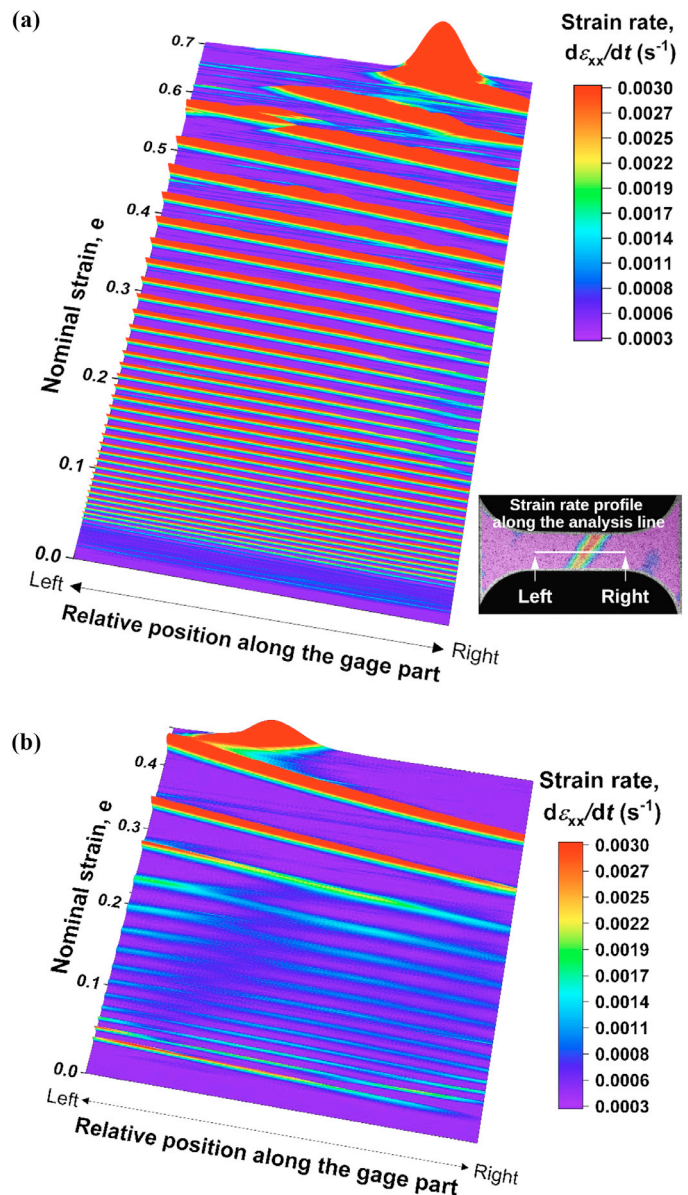


Fig. 3. Overview of changes in local strain rate distribution during tensile deformation for the (a) CG and (b) UFG specimens, obtained by DIC analysis, with the inset of (a) showing how it was measured. The strain rate profiles along the analysis line form the graphs from loading to fracture: a total of 6513 line profiles for the CG specimen and 3949 for the UFG specimen. The magnitude of the local strain rate is represented by different colors based on the key color bar.

patterns with high local strain rate repeatedly appear from right to left on the purple backgrounds with low local strain rate, illustrating the propagation of the localized deformation bands along the gage parts. The high strain rate observed just before fracture corresponds to necking. Although the localized deformation behavior in PLC banding and necking appears similar, it should be noted that the strain-localized region in the PLC bands was able to propagate further due to the remaining strain hardening capacity, unlike in necking. Such local strain hardening behavior will be quantitatively analyzed for both the CG and UFG specimens and discussed later in Fig. 10. Interestingly, the localized deformation behavior was noticeably less pronounced in the UFG specimen than in the CG specimen during the early stage of deformation ($\epsilon = 0.10$ to 0.20). The high strain rate, indicated in red according to the key color bar, was not observed in this stage, suggesting that deformation was more homogeneous in the UFG specimen. For the quantitative evaluation, the true global strain rates were measured as the time derivatives of the true global strains obtained by the DIC analysis, and the results are shown in the Supplementary Material (Fig. S1). The average true global strain rates were approximately 0.00041 s^{-1} for the CG specimen and 0.00049 s^{-1} for the UFG specimen, and they appear in purple according to the key color bar inserted in Fig. 3. This confirms that most regions of the gage parts deformed at substantially lower strain rates than the global values, whereas strain was strongly localized within the PLC bands. In addition, the gaps between wall-like red patterns were wider for the UFG specimen than for the CG specimen, indicating that it took more time for the localized deformation band in the UFG specimen to complete its propagation along the gage part.

To explore the correlation between localized deformation behavior and the global mechanical responses, the nominal stress-strain curves and the local strain rate distributions within a specific strain range were investigated for the CG and UFG specimens. Fig. 4(a) and (b) shows the enlarged nominal stress-strain curves of the CG and UFG specimens, respectively, in a strain range from 0.069 to 0.092 . In this strain range, three serration peaks were observed on the stress-strain curve of the CG specimen, and two serration peaks were observed on the stress-strain curve of the UFG specimen. Serration behavior was more pronounced in the CG specimen, while the UFG specimen exhibited less distinct serration peaks. Points (A, B, C) for the CG specimen and (A', B', C') for the UFG specimen, marked between two neighboring serration peaks, correspond to the DIC maps shown in Fig. 4(c)–(f). Fig. 4(c, d) and (e, f) shows the local strain rate and strain maps for the CG and UFG specimens, respectively, captured at the different global strain levels corresponding to the points (A, B, C) and (A', B', C') on the stress-strain curves shown in Fig. 4(a) and (b). The local strain rate and local strain along the gage part are represented in different colors, as indicated by the respective scale bars. The local banded region with distinctly localized plastic deformation in Fig. 4(c) and (e) corresponds to what is known as a Portevin–Le Chatelier (PLC) band. The propagation of the PLC band along the gage part led to a gradual accumulation of local strain, as shown in Fig. 4(d) and (f). At the first serration peaks on the enlarged stress-strain curves of the CG and UFG specimens (A in Fig. 4(a) and A' in Fig. 4(b)), the former PLC bands were finishing their propagation and annihilating into the grip part, while new PLC bands were simultaneously nucleating at the opposite grip part (A in Fig. 4(c) and A' in Fig. 4(e)). At this point, strain was uniform along the gage part (A in Fig. 4(d) and A' in Fig. 4(f)). During the subsequent monotonic increase in stress (B in Fig. 4(a) and B' in Fig. 4(b)), the new PLC bands were propagating along the gage part (B in Fig. 4(c) and B' in Fig. 4(e)). The PLC band in the CG specimen was clearly banded, exhibiting a high strain rate (0.0029 s^{-1}) shown in red. In contrast, the PLC band in the UFG specimen

was less defined, exhibiting a much lower strain rate (0.0012 s^{-1}) shown in blue, confirming that plastic deformation was less localized in the UFG specimen under the same global strain rate applied ($\sim 0.00077 \text{ s}^{-1}$) in this strain range. The local strain distribution in the CG specimen showed a clear contrast between the area swept by the PLC band and the area not swept (B in Fig. 4(d)), whereas such a clear contrast was not found in the UFG specimen (B' in Fig. 4(f)). As the PLC bands started to annihilate into the grip part (C in Fig. 4(c) and C' in Fig. 4(e)), the local strain accumulated uniformly (C in Fig. 4(d) and C' in Fig. 4(f)), and plastic deformation started to be suppressed along the gage part. Such a suppression of plastic deformation along the gage part caused a rapid increase in stress, forming the serration peaks on the stress-strain curves (C in Fig. 4(a) and C' in Fig. 4(b)). The nucleation, propagation, and annihilation of PLC bands (PLC banding) were observed throughout the entire deformation process in all specimens, including the UFG specimen that exhibited weak strain localization at the early stage of deformation, and such PLC banding in all specimens corresponded perfectly to serration behavior on the global stress-strain curves.

3.4. Strain-hardening behavior in the CG and UFG specimens

Fig. 5(a) and (b) shows the strain-hardening rate ($d\sigma/d\epsilon$, blue) and true stress (σ , red) curves of the CG and UFG specimens, respectively, plotted as a function of true strain (ϵ). Smoothed strain-hardening rates (black-dashed lines) are superimposed on the raw data to estimate the strain-hardening rates of the specimens with the different grain sizes at an identical tensile strain. The strain-hardening rate curve (blue) of the CG specimen (Fig. 5(a)) exhibited severe fluctuation of approximately $\pm 2000 \text{ MPa}$, while that of the UFG specimen (Fig. 5(b)) showed little fluctuation except in the later stage of deformation. The severe fluctuation on the strain-hardening rate curve of the CG specimen corresponded to the serration peaks on its stress-strain curve, whereas the little fluctuation on the strain-hardening rate curve of the UFG specimen, particularly during the early stage of the deformation, corresponded to the less pronounced serration peaks on its stress-strain curve, as shown in Fig. 2. As indicated by the smoothed strain-hardening rates (black-dashed lines), the strain-hardening rate of the CG specimen was approximately 3000 MPa at the early stage of deformation ($\epsilon \sim 0.1$) and gradually decreased to the later stage of deformation ($\epsilon \sim 0.5$), resulting in large uniform elongation. On the other hand, the strain-hardening rate of the UFG specimen was initially as high as over 3000 MPa at the early stage of deformation ($\epsilon \sim 0.1$) but rapidly decreased to below 2000 MPa at the later stage of deformation ($\epsilon \sim 0.37$).

The intersection of the strain-hardening rate curve and the true stress-true strain curve corresponds to the onset of necking, also referred to as plastic instability. Considère's criterion for plastic instability is described by the following formula [40].

$$\sigma \geq d\sigma/d\epsilon \quad (1)$$

To overcome plastic instability and to propagate the strain localized region along the gage part, the strain-hardening rate must be greater than the flow stress ($d\sigma/d\epsilon \geq \sigma$). If the plastic instability (onset of necking) is not overcome, the strain localized region develops into necking, eventually leading to fracture. In this respect, the repeated propagation of the PLC bands (Figs. 3 and 4), i.e., the successive propagation of the strain localized region, can be regarded as a series of processes of overcoming the plastic instabilities. In the subsequent sections, the effect of grain refinement on the serration behavior associated with local PLC banding will be discussed from this perspective.

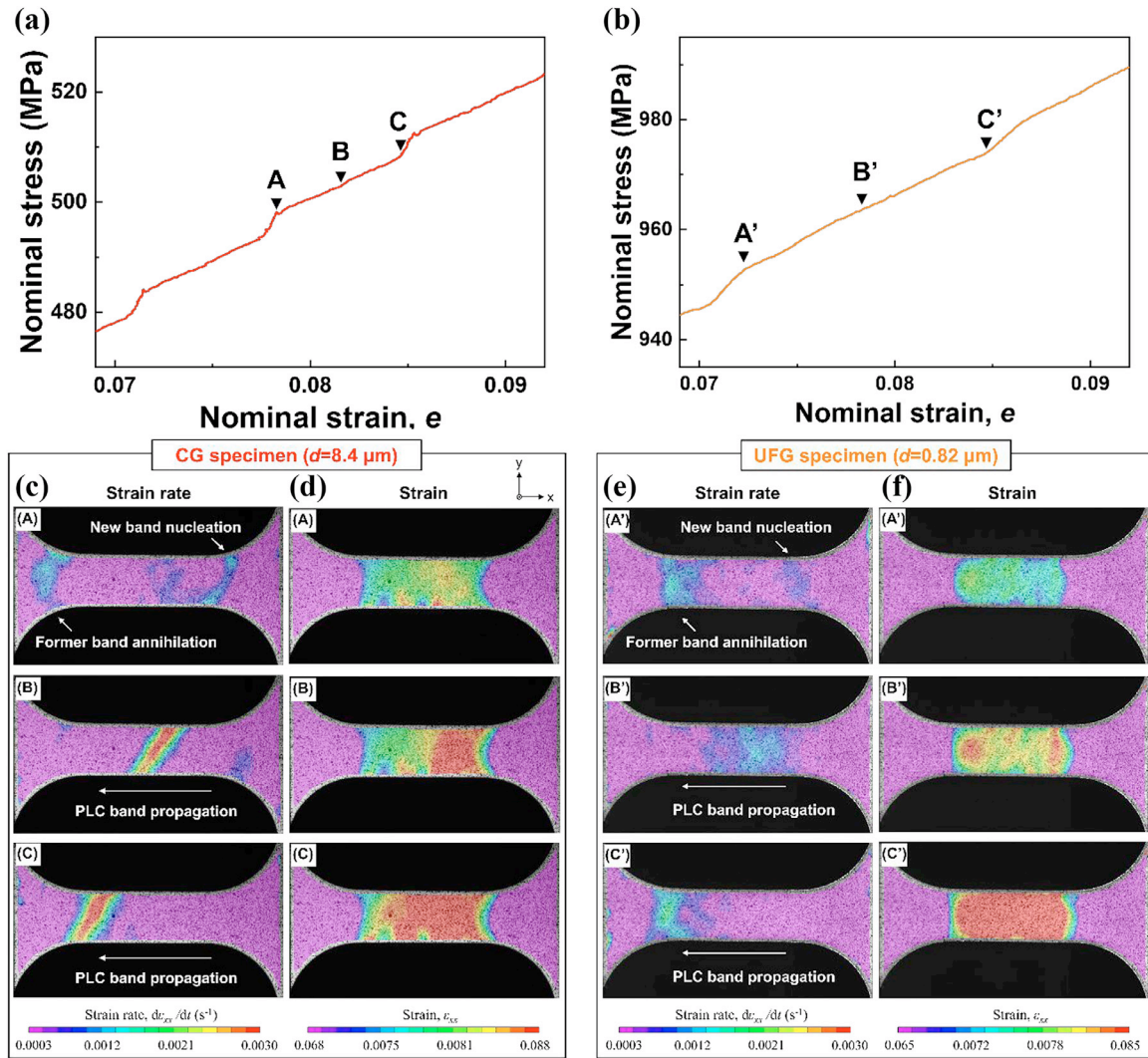


Fig. 4. Enlarged nominal stress-strain curves of the (a) CG and (b) UFG specimens in the strain range from 0.069 to 0.092. The points (A, B, C) for the CG specimen and (A', B', C') for the UFG specimen, marked between two neighboring serration peaks, correspond to the DIC maps shown in (c)–(f). (c) DIC local strain rate maps and (d) strain maps of the CG specimen, and (e) local strain rate maps and (f) strain maps of the UFG specimen, captured at different global strain levels corresponding to the points (A, B, C) and (A', B', C') on the stress-strain curves shown in (a) and (b).

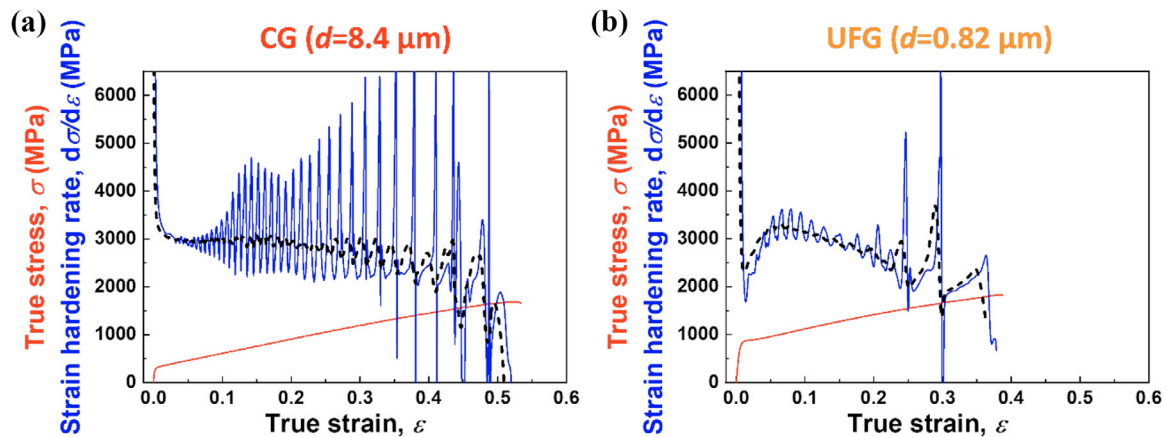


Fig. 5. Strain-hardening rates (blue) and true stress-strain curves (red) of the (a) CG and (b) UFG specimens, plotted as a function of true strain. Smoothed strain-hardening rates, shown as black dashed lines, are superimposed on the raw strain-hardening rates.

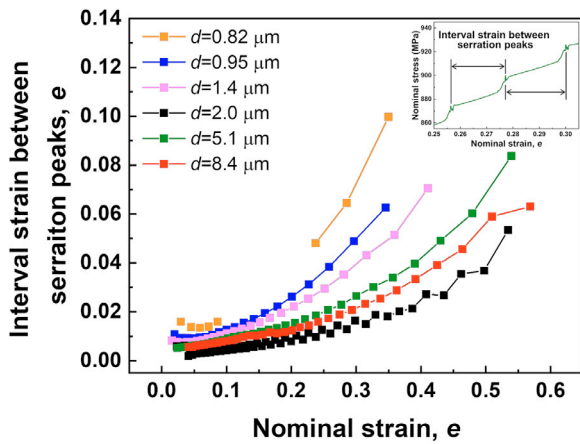


Fig. 6. Interval strain between the serration peaks (period) for the specimens with various grain sizes, plotted as a function of global tensile strain. The period for the UFG specimen in the strain range of 0.10–0.20 was omitted due to its weak serration behavior on the stress-strain curve, as shown in Fig. 2. An inset in the figure shows a representative measurement of the interval strain between the serration peaks for the specimen with a mean grain size of 5.1 μm .

3.5. Effect of grain refinement on serration behavior and local PLC banding

The serration peak on the stress-strain curve was a response to the annihilation of the former PLC band and the formation of a new PLC band (A in Fig. 4(c) and A' in Fig. 4(e)) in the tensile specimen. Therefore, the interval strain between the serration peaks on the stress-strain curve corresponds to the difference in global tensile strain required to complete one cycle of the PLC banding along the tensile specimen. Fig. 6 shows the interval strain between the serration peaks on the stress-strain curves of the specimens with various grain sizes, plotted as a function of nominal strain. The interval strain between the serration peaks was obtained from the nominal stress-strain curves of the various grain-sized specimens (Fig. 2), and will be termed the period hereafter. Since the UFG specimen exhibited weak serration behavior during the early stage of deformation (Fig. 2), the period for the UFG specimen in a strain range from 0.10 to 0.20 was omitted to accurately evaluate the effect of grain refinement on the period. It was observed that the periods increased as deformation progressed for all specimens. At the early stage of deformation ($e \sim 0.1$), the periods for the specimens with various grain sizes were consistently small, ranging from 0.004 (black, the specimen with a mean grain size of 2.0 μm) to 0.016 (yellow, UFG specimen). However, as deformation progressed, the periods increased significantly, ranging from 0.053 (black, the specimen with a mean grain size of 2.0 μm) to 0.100 (yellow, UFG specimen) at the later stage of deformation. Interestingly, it was also evident that the period increased with grain refinement, except for the specimen with a mean grain size of 2.0 μm . The period of this specimen will be addressed later in Fig. 7(c).

The increase in the period with grain refinement indicates that more time (or a larger global tensile strain) was required for a PLC band to complete its propagation along the tensile specimen. To systematically understand the changes in PLC banding behavior with grain refinement, the PLC band velocity in specimens with various grain sizes was analyzed using the DIC technique. Fig. 7(a) and (b) shows the representative analysis performed on the specimen with a mean grain size of 5.1 μm , for measuring the PLC band velocity. In Fig. 7(a), two points (P1 and P2) were set in the DIC strain rate map, and a digital extensometer was placed between the two points to track the change in distance between the two points during deformation. The changes in local strain rate at

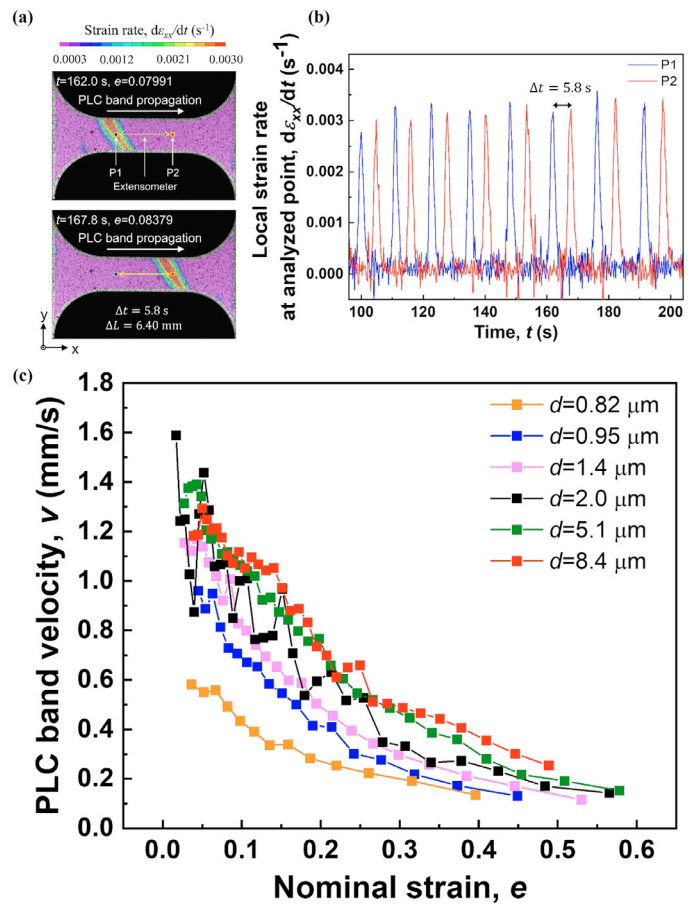


Fig. 7. (a) In the DIC strain-rate map of the specimen with a mean grain size of 5.1 μm , two points (P1 and P2) were set in the gage part, and a non-contact digital extensometer was placed between them. (b) The profile of the local strain rate at the two points from 95 s to 205 s was extracted. The sharp peaks corresponded to the passage of the PLC bands through the two points, and the time difference (Δt) corresponded to the duration taken by the PLC band to propagate from P1 to P2. (c) The PLC band velocity for the specimens with various mean grain sizes, plotted as a function of nominal strain.

points P1 and P2 (Fig. 7(a)) were investigated in Fig. 7(b). Sharp peaks on the strain rate-time curve appeared periodically whenever the PLC band passed through the analyzed points, so the time difference between two adjacent strain rate peaks corresponded to the time taken for the PLC band to propagate from P1 to P2. The PLC band velocity was calculated as the distance propagated by the PLC band divided by the time taken for the propagation. The measurements were performed for all specimens throughout the entire deformation process, and the results are plotted as a function of global tensile strain in Fig. 7(c). Contrary to the increase in the period with grain refinement shown in Fig. 6, the PLC band velocity decreased with deformation for all specimens. At the early stage of deformation ($e \sim 0.1$), the PLC band velocity for the various grain-sized specimens was notably fast, ranging from 0.43 mm s^{-1} (yellow, UFG specimen) to 1.12 mm s^{-1} (red, CG specimen). However, it decreased substantially, reaching as low as 0.11 mm s^{-1} (yellow, UFG specimen) and 0.25 mm s^{-1} (red, CG specimen) at the later stage of deformation. The decrease in PLC band velocity as deformation progressed can be attributed to the decline in the strain-hardening rate during the tensile deformation (Fig. 5), which requires more plastic deformation, i.e., more deformation time, for the local region within the PLC band to be strain hardened.

Moreover, it was found that the PLC band velocity at an identical global tensile strain decreased with grain refinement. The spec-

imen with a mean grain size of $2.0\ \mu\text{m}$ (black dots and line), which deviated from the overall trend in the period-grain size relationship (Fig. 6), aligned with the tendency of decreasing PLC band velocity with grain refinement in this case. This was because the PLC band in the specimen with a mean grain size of $2.0\ \mu\text{m}$ atypically nucleated near the gage center and propagated sequentially toward the left and right halves of the gage part, forming two serration peaks while sweeping the entire gage part once. At a strain level of $e \sim 0.1$, the PLC band velocity in the CG specimen was $1.12\ \text{mm s}^{-1}$, whereas it was $0.43\ \text{mm s}^{-1}$ in the UFG specimen, less than half of that in the CG specimen. At a strain level of $e \sim 0.3$, the PLC band velocity in the CG specimen was $0.49\ \text{mm s}^{-1}$, whereas it was $0.20\ \text{mm s}^{-1}$ in the UFG specimen, also less than half of that in the CG specimen. The decrease in PLC band velocity with grain refinement can be attributed to the necessity of a higher strain-hardening rate to prevent a strain-localized region (PLC band) from progressing into necking and to propagate it into the adjacent area, as discussed in Eq. (1). Since the flow stress of the UFG specimen was higher than that of the CG specimen at an identical tensile strain (Fig. 2), the local strain-hardening rate required to prevent necking development in the UFG specimen should be greater than that in the CG specimen. That is, the local region within the PLC band of the UFG specimen must experience a greater stress increment compared to the CG specimen at an identical global tensile strain. This required more deformation time under the same applied global strain rate and led to a decrease in PLC band velocity with grain refinement (Fig. 7(c)). The slower propagation of PLC band with grain refinement will be discussed in detail later in Fig. 10 in relation to local strain-hardening behavior.

In order to further investigate the effect of grain refinement on the PLC banding in association with the local strain-hardening behavior, the increase in local strain caused by local PLC banding was evaluated in the specimens with various mean grain sizes. Fig. 8(a) shows the representative analysis performed on the specimen with a mean grain size of $5.1\ \mu\text{m}$. An analyzed point was set in the gage part, and changes in local strain rate (blue, $d\epsilon_{xx}/dt$) and local strain (red, ϵ_{xx}) during the middle stage of deformation were investigated. The local strain rate was close to zero when the PLC band was beyond the analyzed point, but it increased rapidly, forming a peak during the passage of the PLC band on the analyzed point. The local strain increased in a stepwise manner, with little increase while the PLC band was beyond the analyzed point and rapid increase as the PLC band passed over it. The increase in local strain caused by local PLC banding was obtained by measuring the height of the step in the local strain profile at the analyzed point, as indicated in Fig. 8(a). By such a measurement, the increase in local strain caused by local PLC banding was investigated in all specimens over the entire deformation, and the result is plotted as a function of nominal strain in Fig. 8(b). For all specimens, the increase in local strain caused by local PLC banding increased with deformation. At the early stage of deformation ($e \sim 0.1$), the increase in local strain caused by local PLC banding was relatively small, commonly around 0.01. However, as deformation progressed, the increase in local strain caused by local PLC banding exhibited a significant increase, reaching levels from 0.09 (red, CG specimen) to as high as 0.21 (pink, the specimen with a mean grain size of $1.4\ \mu\text{m}$) at the later stage of deformation. As mentioned in Section 3.2 (Fig. 2), post-uniform elongation was quite small in all specimens. This was because such huge strain localization at the later stage of deformation exceeded the remaining strain-hardening capacity of the material, and this strain localization then developed into necking shortly followed by fracture. Interestingly, it was also found that the increase in local strain caused by local PLC banding rose with grain refinement. As discussed in the former paragraph (Fig. 7(c)), the local region within

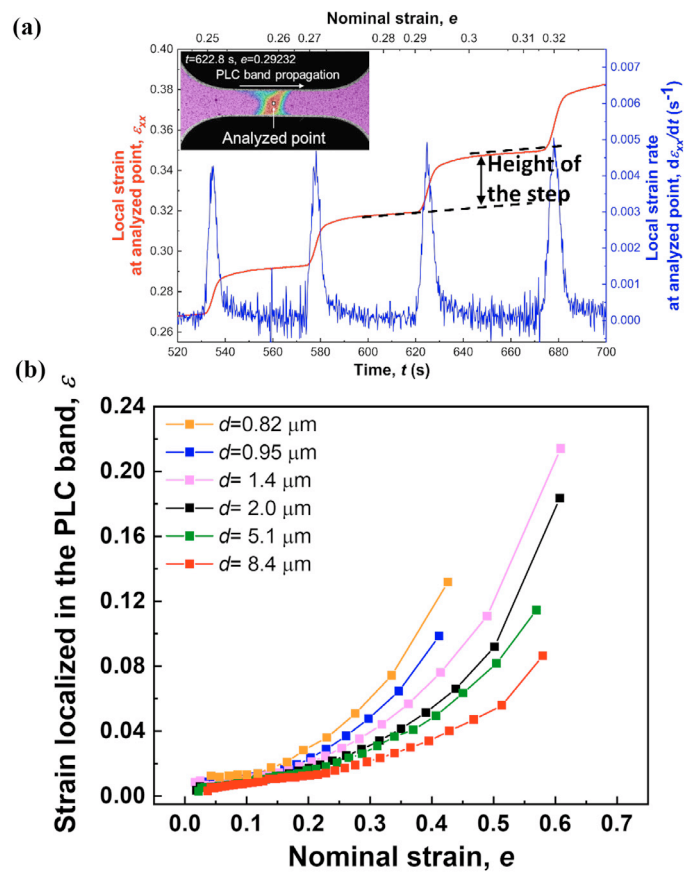


Fig. 8. (a) A representative measurement of the increase in local strain caused by local PLC banding, performed on the profile of the local strain and strain rate in the specimen with a mean grain size of $5.1\ \mu\text{m}$. The increase in local strain caused by local PLC banding corresponded to the height of the step. (b) The increase in local strain caused by local PLC banding for the specimens with various mean grain sizes, plotted as a function of nominal strain.

the PLC band of the UFG specimen required greater strain hardening than that of the CG specimen to prevent necking and to enable the propagation of the strain-localized region (PLC band). Therefore, the increase in local strain caused by PLC banding after grain refinement (Fig. 8(b)) can be consistently understood in terms of the greater strain hardening required to suppress necking and sustain the propagation of the PLC band. The local strain hardening will be quantitatively analyzed for both the CG and UFG specimens and discussed later in Fig. 10.

Next, the homogeneity (or heterogeneity) of plastic deformation along the gage part was evaluated to identify the cause of changes in serration behavior, particularly changes associated with PLC banding, with grain refinement. Fig. 9(a) and (b) shows the representative analyses performed on the CG and UFG specimens, respectively. The profiles of local strain (ϵ_{xx}) and local strain rate ($d\epsilon_{xx}/dt$) of the CG and UFG specimens at the early stage of the deformation ($t = 310\text{--}510\ \text{s}$) are enlarged. A strain rate of zero ($d\epsilon_{xx}/dt = 0$) is drawn in the graph as green-dashed line as a reference to indicate the absence of plastic deformation. As discussed in Fig. 8(a), the rapid increase in local strain and periodic sharp peaks in local strain rate corresponded to the duration while the PLC band was passing through the analyzed point. In contrast, the slow increase in local strain (the relatively flat part of the step) and the low local strain rate between the periodic sharp peaks corresponded to the duration while the PLC band was beyond the analyzed point. Interestingly, the local strain in the CG specimen (Fig. 9(a)) increased in a clear stepwise manner, whereas it in-

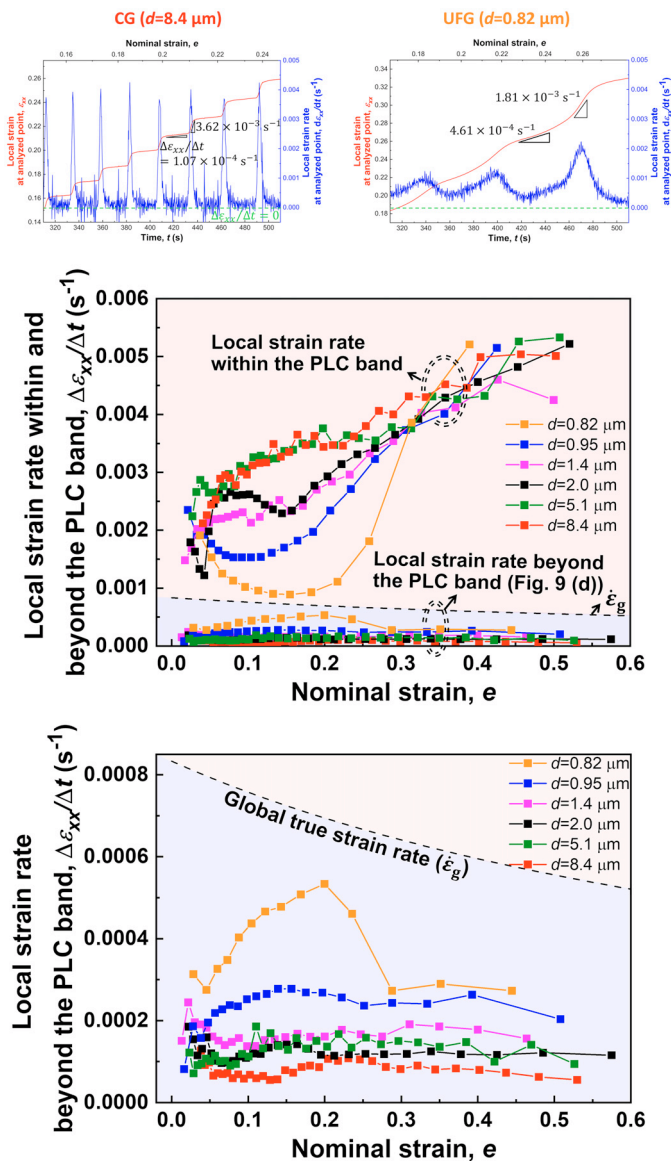


Fig. 9. Enlarged profiles of the local strain and strain rate for the (a) CG and (b) UFG specimens at the early stage of the deformation from 310 s to 510 s. The slopes of the local strain-time curve are indicated by black triangles ($\Delta \epsilon_{xx} / \Delta t$). A strain rate of zero ($\Delta \epsilon_{xx} / \Delta t = 0$) is drawn in the graph as a green-dashed line, serving as a reference to indicate absence of plastic deformation. (c) Local strain rate within the PLC band ($\dot{\epsilon}_{\text{within}}$) and beyond the PLC band ($\dot{\epsilon}_{\text{beyond}}$) for the various grain-sized specimens, plotted as a function of global tensile strain. The black-dashed line indicates the global true strain rate ($\dot{\epsilon}_g$). (d) Enlarged plot of the local strain rate beyond the PLC band for the various grain-sized specimens.

creased in a less distinct stepwise manner in the UFG specimen (Fig. 9(b)). When the PLC band was within the analyzed point, the slope of the local strain increment ($\dot{\epsilon}_{\text{within}}: \Delta \epsilon_{\text{within}} / \Delta t_{\text{within}}$) in the UFG specimen was $1.81 \times 10^{-3} \text{ s}^{-1}$, which was two times lower than that in the CG specimen ($3.62 \times 10^{-3} \text{ s}^{-1}$). On the other hand, when the PLC band was beyond the analyzed point, the slope of the local strain increment in the UFG specimen ($\dot{\epsilon}_{\text{beyond}}: \Delta \epsilon_{\text{beyond}} / \Delta t_{\text{beyond}}$) was $4.61 \times 10^{-4} \text{ s}^{-1}$, which was approximately four times higher than that in the CG specimen ($1.07 \times 10^{-4} \text{ s}^{-1}$). The local strain rate between the strain rate peaks in the CG specimen overlapped with the zero strain rate (green-dashed line), whereas in the UFG specimen, it was above the zero strain rate. These results indicate that the UFG specimen experienced some plastic deformation even in the region beyond

the PLC band, whereas the CG specimen exhibited strongly suppressed plastic deformation in this region, with most of the plastic deformation occurring within the PLC band. Such a difference in the distribution of local strain between the CG and UFG specimens strongly suggests that the homogeneity of plastic deformation along the gage part was dependent on the grain size of the specimen. To evaluate the grain size dependence on the homogeneity of plastic deformation in more detail, the slope of the local strain increment was investigated for all specimens both when the PLC band was within the analyzed point ($\dot{\epsilon}_{\text{within}}$) and when it was beyond the analyzed point ($\dot{\epsilon}_{\text{beyond}}$) throughout the entire deformation. The results are plotted as a function of nominal strain in Fig. 9(c). $\dot{\epsilon}_{\text{within}}$ and $\dot{\epsilon}_{\text{beyond}}$ refer to the local strain rate within the PLC band and the local strain rate beyond the PLC band, respectively, and are represented with red and blue backgrounds in the figure. In Fig. 9(c), the global true strain rate ($\dot{\epsilon}_g$) is also drawn as a black-dashed line to evaluate the homogeneity of plastic deformation along the gage part. If every point in the gage part deforms consistently with the global true strain rate applied at a certain stage of deformation, it indicates that the deformation is spatially homogeneous throughout the entire gage part. The global true strain rate was calculated as:

$$\dot{\epsilon}_g = \dot{\epsilon}_0 \times l_0 / l \quad (2)$$

where $\dot{\epsilon}_0$, l_0 and l are the initially applied strain rate, initial gage length, and gage length at a certain strain level, respectively. It should be noted that the average global true strain rates (Fig. S1), measured as the time derivative of the global true strain obtained by DIC analysis, are lower than the global true strain rate ($\dot{\epsilon}_g$ shown in Fig. 9(c) and (d)), as the values in Fig. S1 include the periods during which the PLC band propagates outside the gage part, appearing as periodic downspikes and reducing the overall averages. For all specimens, the local strain rate within the PLC band remained higher than the global true strain rate ($\dot{\epsilon}_g$), while the local strain rate beyond the PLC band remained lower throughout the entire deformation, confirming that plastic deformation was localized within the PLC band regardless of grain size. With grain refinement from 8.4 μm (red, CG specimen) to 1.4 μm (pink), the local strain rate within the PLC band generally decreased. During the early stage of deformation ($e = 0.10$ – 0.20), the local strain rate within the PLC band ranged from 0.00213 s^{-1} (pink, 1.4 μm grain-sized specimen) to 0.00365 s^{-1} (red, CG specimen), which was much higher than the global true strain rate of 0.00072 s^{-1} in this strain range, indicating that plastic deformation was strongly localized within the PLC band. However, with further grain refinement to the UFG specimen (yellow), the local strain rate within the PLC band rapidly decreased, particularly during the early stage of deformation. For the UFG specimen, the local strain rate within the PLC band dropped to 0.00089 s^{-1} at $e = 0.15$, which was quite close to the global true strain rate of 0.00072 s^{-1} at the same tensile strain. To further investigate the homogeneity (or heterogeneity) of plastic deformation along the gage part, the local strain rate beyond the PLC band, which is represented with blue background in Fig. 9(c), is enlarged in Fig. 9(d). Interestingly, the local strain rate beyond the PLC band was the reflection of that within the PLC band across the global true strain rate. In the early stage of deformation ($e = 0.10$ – 0.20), specimens with grain sizes ranging from CG (red) to 4 μm (pink) showed much lower local strain rates beyond the PLC band compared to the global true strain rate, indicating strong suppression of plastic deformation beyond the PLC band. However, with further grain refinement to the UFG specimen, the strain rate beyond the PLC band increased rapidly, particularly in the early stage of deformation. The local strain rate beyond the PLC band for the UFG specimen reached 0.00053 s^{-1} at $e = 0.20$, which was quite close to the global true strain rate of 0.00070 s^{-1} at the same strain. For the UFG specimen, the lo-

cal strain rates both within (Fig. 9(c)) and beyond the PLC band (Fig. 9(d)) were close to the global true strain rate, indicating that the entire gage part deformed more homogeneously (or less heterogeneously) with grain refinement. As shown in Fig. 2, serration behavior in the UFG specimen was weak during the early stage of the deformation ($\epsilon = 0.10$ – 0.20), while the CG specimen exhibited obvious serration behavior over the same tensile strain range. In the CG specimen, plastic deformation rarely occurred beyond the PLC band (Fig. 9(d)), possibly because dislocations beyond the PLC band were pinned by carbon atoms. Conversely, the flow stress level of the UFG specimen was over 1000 MPa, even during the early stage of the deformation ($\epsilon = 0.10$ – 0.20), which was approximately 450 MPa higher than that of the CG specimen at the same tensile strain range. Such a high flow stress level in the UFG specimen enabled the dislocations to be de-pinned from carbons, resulting in the tensile specimen deforming more homogeneously along the entire gage part (Fig. 9(c) and (d)) and leading to the weak serration behavior observed in the UFG specimen (Fig. 2).

3.6. Local strain-hardening behavior of the CG and UFG specimens revealed through the simultaneous application of in-situ synchrotron XRD measurement and DIC analysis

The propagation of the PLC band was examined in terms of a process of overcoming the plastic instability condition (necking), and all findings in the previous sections consistently indicated that more strain hardening was required with grain refinement to overcome necking and continue the propagation of the PLC band. Finally, in the current section, *in-situ* synchrotron XRD measurement was carried out during the tensile deformation to evaluate the local strain-hardening behavior of the CG and UFG specimens in association with microstructure evolution, such as dislocation density and stacking fault probability, with a high temporal resolution of one second. By applying *in-situ* synchrotron XRD measurement in combination with DIC analysis, the microstructure evolution and the consequent change in lattice strain in the local area (beam position) can be obtained with respect to the propagation of the PLC band.

The results obtained from the XRD measurement and DIC analysis were synchronized precisely and are shown in Fig. 10. Fig. 10(a, b) and (c, d) shows the DIC maps of the CG and UFG specimens, respectively, captured at different deformation times around 500 s, during which the PLC band were propagating near the beam position. The local strain rate and strain maps of the CG (1, 2, 3) and UFG (1', 2', 3') specimens corresponded to the XRD measurement results shown later in Fig. 10(g, m) and (h, n), respectively. At (1) in Fig. 10(a), the PLC band nucleated near the gage center and propagated to the left. Since the PLC band did not pass the beam position, the local strain at the beam position was not yet accumulated ((1) in Fig. 10(b)). At (1') in Fig. 10(c), the PLC band started to propagate to the right from the left end of the gage part, and the local strain was uniformly distributed along the gage part, including the beam position ((1') in Fig. 10(d)). At (2) in Fig. 10(a) and at (2') in Fig. 10(c), the PLC bands were passing through the beam position, and the local strains at the beam position started to be accumulated ((2) in Fig. 10(b) and (2') in Fig. 10(d)). At (3) in Fig. 10(a) and at (3') in Fig. 10(c), the PLC bands left the beam position, and the local strains at the beam position were completely accumulated ((3) in Fig. 10(b) and (3') in Fig. 10(d)).

Next, changes in lattice strain (red), dislocation density (blue) and stacking fault probability (green) at the XRD beam position, as well as global tensile stress (black), during the tensile deformation of the CG and UFG specimens are plotted as a function of nominal strain in Fig. 10(e) and (f), respectively. The deformation time corresponding to the nominal strain is also shown on the upper x-axis. Lattice strain (or elastic strain) was calculated

as the change in interplanar spacing divided by the initial interplanar spacing of a selected diffraction peak. As stress is linearly proportional to lattice strain through the elastic modulus, according to the well-known Hooke's law, the lattice strain discussed in this study represents the local elastic stress at the beam position. In this study, the {200} peak was selected because it exhibited the second-highest diffraction intensity and the largest peak shift among all diffraction peaks of the present material with an FCC structure [8,41]. Dislocation density was obtained using the Williamson-Hall method, which involves analyzing the FWHMs of the diffraction peaks. Stacking fault probability describes how frequently stacking faults occur on the {111} planes in the FCC matrix [42–44]. It can be understood as the statistical fraction of atomic layers where the ideal stacking sequence is disrupted. In high-Mn austenitic steels with an FCC structure, stacking faults introduced on the {111} planes cause characteristic shifts in diffraction peak positions, depending on the crystallographic plane. According to Warren's theory [44], stacking faults in the FCC matrix shift diffraction peaks either to higher or lower angles depending on the $|h + k + l|$ for each (hkl) subplane. For example, the formation of stacking faults causes a higher-angle shift in the {111} peak and a lower-angle shift in the {222} peak, as a net result of contributions from each subplane. A detailed explanation of the evaluation methods for dislocation density and stacking fault probability is provided in the Supplementary Material. Since the {111} and {222} planes are subject to the same strain tensor during deformation, the changes in measured interplanar spacing due to stacking faults can be used to estimate the stacking fault probability as follows [45,46]:

$$P_{\text{SF}} = 32\pi / (3\sqrt{3}) \times (d_{222}/d_{222}^0 - d_{111}/d_{111}^0) \quad (3)$$

where P_{SF} is the stacking fault probability, d_{222} and d_{111} are the measured interplanar spacings of {222} and {111} planes, respectively, and d_{222}^0 and d_{111}^0 are their initial interplanar spacings in the undeformed state.

Meanwhile, Balogh et al. [42] reported that the formation of deformation twins leads to symmetric peak broadening in the diffraction profiles of FCC crystals. The measurement system itself can also cause peak broadening in diffraction profiles due to factors such as the X-ray source size, detector resolution, and optical alignment, a phenomenon known as instrumental broadening [47]. However, as both the UFG and CG specimens were measured using the same equipment and analysis method, this effect is expected to be comparable between them. In the present study, peak broadening caused by deformation twins and instrumental effects was not separated in the FWHM analysis, which may have led to an overestimation of dislocation density. Although twins were not individually resolved, their effect is qualitatively reflected in the trend in stacking fault probability, as deformation twins can be regarded as a series of stacking faults formed on successive {111} planes [38].

As shown in Fig. 10(e) and (f), the nominal stress-nominal strain curves (black) show that the flow stress level was much higher in the UFG specimen than in the CG specimen. Serration behavior was observed on the nominal stress-nominal strain curve of the CG specimen throughout its entire deformation, while the UFG specimen showed relatively weak serration behavior, especially during its early and mid-stages of deformation. On the lattice strain-nominal strain curves of the CG and UFG specimens (red), lattice strain in both specimens increased as the deformation proceeded. Notably, the lattice strain was much higher in the UFG specimen than in the CG specimen at an identical nominal strain. Additionally, a periodic increase in lattice strain was observed on the curves of both specimens. The increase in lattice strain was larger in the UFG specimen than in the CG specimen. On the dislocation density-nominal strain curves of the CG and UFG specimens

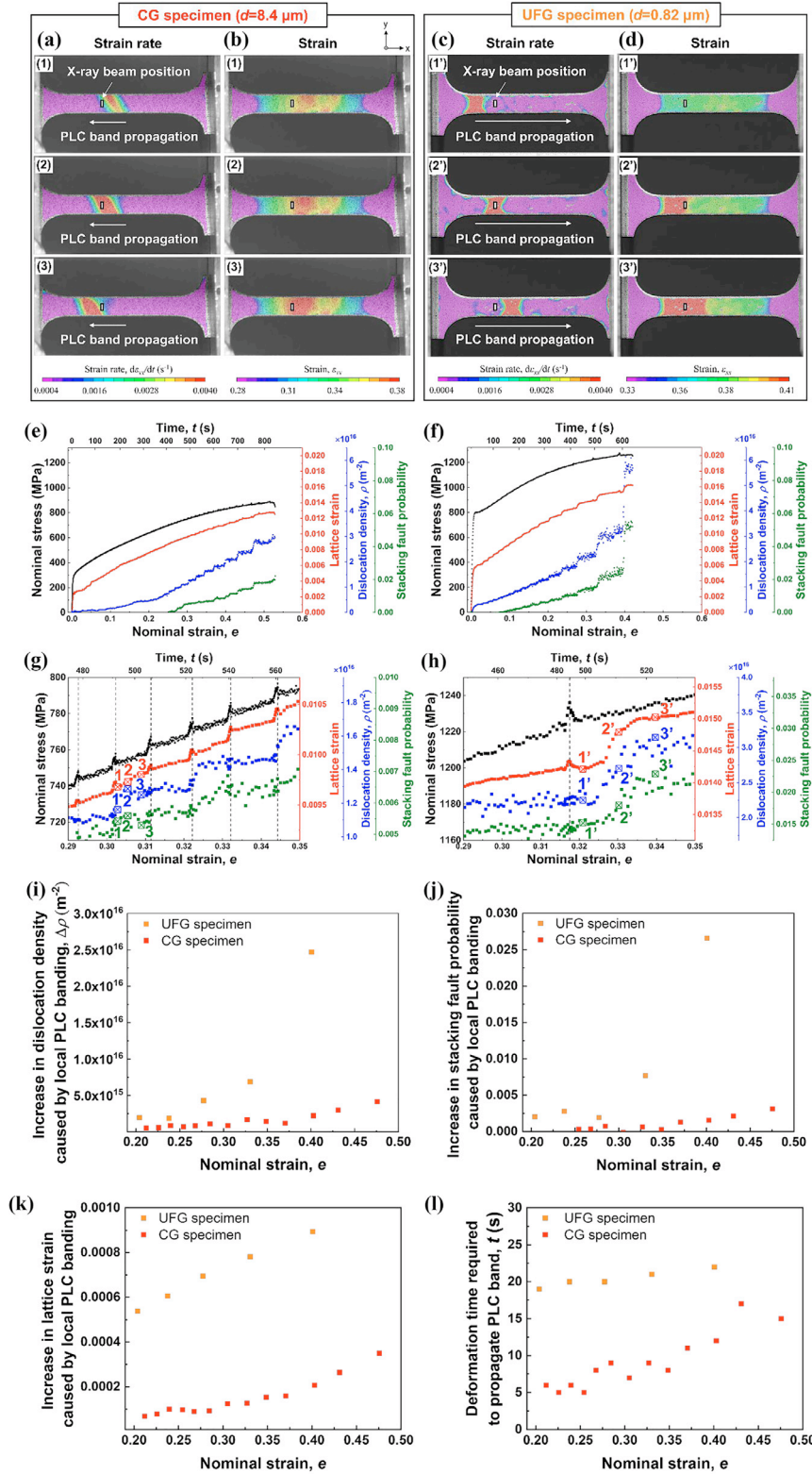


Fig. 10. (a) DIC local strain rate maps and (b) strain maps of the CG specimen, and (c) local strain rate maps and (d) strain maps of the UFG specimen, captured at different deformation times around 500 s during the propagation of the PLC bands near the beam position. The sets of DIC maps numbered (1, 2, 3) for the CG specimen and (1', 2', 3') for the UFG specimen are directly associated with the XRD results marked with the same numbers in (g, m) and (h, n), respectively. Changes in local lattice strain (red, first right y-axis), dislocation density (blue, second right y-axis), and stacking fault probability (green, third right y-axis) at the XRD beam position, and nominal stress (black, left y-axis) are plotted as a function of nominal strain for the (e) CG and (f) UFG specimens, respectively. Enlarged curves for the (g) CG and (h) UFG specimens are shown within the nominal strain range of $e = 0.29$ to 0.35 . Black-dashed lines are drawn at the nominal strain levels when serration peaks appeared on the curves. The increases in (i) dislocation density, (j) stacking fault probability, and (k) lattice strain caused by local PLC banding, as well as (l) the deformation time required to propagate PLC band in the CG and UFG specimens, are plotted as functions of nominal strain. Local strain-hardening rates (blue) and local true stress curves (red) of the (m) CG and (n) UFG specimens, plotted as functions of true strain. The local area in the inset figures corresponds to exactly to the fixed X-ray beam position. The points $\blacksquare$ (1, 2, 3) and $\blacksquare$ (1', 2', 3') on the curves correspond to the deformation times at which the DIC images of (1, 2, 3) and (1', 2', 3') in (a)–(d) were captured.

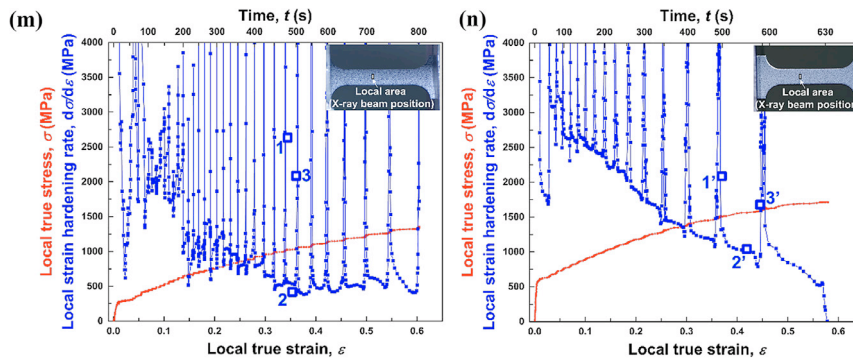


Fig. 10. Continued

(blue), dislocation density was larger in the UFG specimen than in the CG specimen at an identical nominal strain. Dislocation density in both specimens increased in a stepwise manner, showing little increase during most of the deformation but a significant increase at certain nominal strains (or deformation times). On the stacking fault probability-nominal strain curves of the CG and UFG specimens (green), the overall trend was similar to that of dislocation density. From the middle stages of deformation ($e > 0.25$), the stacking fault probability remained nearly constant during most of the subsequent deformation, and increased significantly at specific nominal strains (or deformation times). On the other hand, stacking faults were not detected during the early stages of deformation, particularly in the CG specimen. This may be attributed to the roughly thousand times fewer grains within the beam path in the CG specimen compared to the UFG specimen, which resulted in fewer {111} or {222} planes satisfying the Bragg condition. However, grain subdivision during deformation increased the number of planes satisfying the Bragg condition and enabled the quantification of stacking fault probability. From the middle stages of deformation, the UFG specimen exhibited a higher stacking fault probability than the CG specimen at an identical nominal strain, though it still remained below 0.03 throughout most of its deformation.

To further investigate the changes in lattice strain and deformation microstructure in relation to PLC banding, the curves of the CG and UFG specimens during the same nominal strain range ($e = 0.29$ to 0.35) are enlarged in Fig. 10(g) and (h), respectively. Black-dashed lines are drawn at the nominal strain levels at which serration peaks appeared on the nominal stress-nominal strain curves. Both the CG and UFG specimens showed that the serration peaks on the nominal stress-nominal strain curves (black) and lattice strain peaks on the lattice strain-nominal strain curves (red) formed simultaneously, indicating that both specimens experienced abrupt elastic deformation throughout the entire tensile specimens including the beam position. The points (1, 2, 3) and (1', 2', 3') on the curves of the CG and UFG specimens, respectively, correspond to the (1, 2, 3) and (1', 2', 3') of the DIC maps previously shown in Fig. 10(a)–(d). Comparing the DIC maps with the enlarged curves of the CG specimen, it was found that dislocation density noticeably increased (1 to 3 in Fig. 10(g)) as the PLC band passed through the beam position ((1)–(3) in Fig. 10(a)), during which stacking fault probability showed a subtle decrease by 0.0001 (1 to 3 in Fig. 10(g)) as part of an overall step-wise increasing trend. As a result of such accumulation of crystal defects, lattice strain increased (1 to 3 in Fig. 10(g)). In the CG specimen, the PLC band was observed to nucleate near the gage center and propagate alternately to the left and to the right. As the beam position was relatively on the left side, rightward propagation could not be detected. For this rea-

son, no change in dislocation density and stacking fault probability was observed during rightward propagation, such as between the serration peak at $e = 0.311$ and the subsequent serration peak at $e = 0.322$ in Fig. 10(g). For the UFG specimen, dislocation density (1' to 3' in Fig. 10(h)) and stacking fault probability (1' to 3' in Fig. 10(h)) were barely increasing before the PLC band passed through the beam position ((1') in Fig. 10(c)), and as a result, lattice strain (1' to 3' in Fig. 10(h)) showed little increase. As the PLC band passed through the beam position ((1') to (3') in Fig. 10(c)), dislocation density (1' to 3' in Fig. 10(h)) and stacking fault probability (1' to 3' in Fig. 10(h)) drastically increased, resulting in a significant increase in lattice strain (1' to 3' in Fig. 10(h)) within the propagating PLC band. After the PLC band of the UFG specimen completely left the beam position ((3') in Fig. 10(c)), dislocation density (3' in Fig. 10(h)), stacking fault probability (3' in Fig. 10(h)), and lattice strain (3' in Fig. 10(h)) increased slowly again. It is noteworthy that the increases in dislocation density (1' to 3' in Fig. 10(h)) and stacking fault probability (1' to 3' in Fig. 10(h)) were both more pronounced in the UFG specimen than in the CG specimen (1 to 3 and 1 to 3 in Fig. 10(g)), which consequently led to greater increase in lattice strain in the UFG specimen (1' to 3' in Fig. 10(h)) compared to that in the CG specimen (1 to 3 in Fig. 10(g)). This suggests that local PLC banding induced greater strain hardening in the UFG specimen than in the CG specimen.

Meanwhile, as noted earlier in Figs. 3(b), 4(e), 9(d) and 10(c), plastic deformation was also occurring to some extent beyond the PLC band in the UFG specimen. On the other hand, the dislocation density and stacking fault probability rarely increased in both the CG (Fig. 10(e, g)) and UFG (Fig. 10(f, h)) specimens when the PLC band was beyond the beam position, during which the slope was rather flat. In this context, it appears plausible that the plastic deformation occurring beyond the PLC band in the UFG specimen was not caused by the increase in dislocation density and stacking fault probability, but rather by dislocation slip (either perfect or leading partial), which was likely facilitated by a higher flow stress level that effectively enabled the de-pinning of dislocations from carbon atoms during the thermal activation process.

To quantitatively compare the local strain-hardening behavior in the propagating PLC band in both specimens, the increases in dislocation density, stacking fault probability, and lattice strain caused by local PLC banding are plotted in a strain range from 0.19 to 0.50 in Fig. 10(i), (j), and (k), respectively. Within this strain range, changes in dislocation density, stacking fault probability, and lattice strain can be clearly observed, enabling a precise comparison of local strain-hardening behavior between the UFG and CG specimens. Such changes in the crystal defects and lattice strain caused by local PLC banding were evaluated as follows: for the

CG specimen, the increase in crystal defects was the difference between $\square 1$ and $\square 3$ (or $\square 1$ and $\square 3$) in Fig. 10(g), and the increase in lattice strain was the difference between $\square 1$ and $\square 3$ in Fig. 10(g). The same measurement was applied to the UFG specimen as well. As shown in Fig. 10(i) and (j), the increase in dislocation density and stacking fault probability caused by local PLC banding rose as the deformation progressed in both the CG and UFG specimens. Interestingly, it was also found that the increase in dislocation density and stacking fault probability was larger in the UFG specimen than in the CG specimen at an identical nominal strain. Such a larger increase in the crystal defects in the UFG specimen led to a greater rise in local strain caused by PLC banding (Fig. 8(b)). Fig. 10(k) shows that the increase in lattice strain caused by local PLC banding also rose as the deformation progressed in both the CG and UFG specimens. Notably, it was also found that the increase in lattice strain was larger in the UFG specimen than in the CG specimen at an identical nominal strain. The larger increases in the crystal defects (Fig. 10(i) and (j)) and lattice strain (Fig. 10(k)) in the UFG specimen confirm that greater local strain hardening was required compared to the CG specimen to prevent the strain-localized region from developing into necking and to sustain PLC band propagation. Furthermore, the deformation time required to propagate the PLC band was also measured for both specimens and is plotted in Fig. 10(l). For example, in the case of the CG specimen, it was determined as the time difference between $\square 1$ and $\square 3$ in Fig. 10(g), and the same measurement was applied to the UFG specimen as well. This propagation time corresponds to the time taken for the local region to complete strain hardening. The time taken to complete local strain hardening increased with deformation in both specimens. The UFG specimen required more time than the CG specimen, but the time gap between them narrowed and eventually converged in the later stages of deformation. Grain refinement increased the time required to complete local strain hardening, which appeared at the specimen scale as a decrease in PLC band velocity in the early and middle stages of deformation (Fig. 7(c)). As deformation progressed, the difference in the time required to complete local strain hardening became smaller (Fig. 10(l)), and it appeared at the specimen scale as the PLC band velocity in all specimens converged to a common low value during the later stages of deformation (Fig. 7(c)).

Finally, the local strain hardening rate was evaluated for both the CG and UFG specimens to understand local PLC banding in terms of a process overcoming plastic instability condition (necking). Fig. 10(m) and (n) show the local strain-hardening rate ($d\sigma/d\varepsilon$, blue) and local true stress (σ , red) curves of the CG and UFG specimens as a function of local true strain (ε), respectively. The deformation time corresponding to the true strain is also shown on the upper x-axis. Here, the local area corresponds exactly to the fixed X-ray beam position, as indicated in the inset figures of Fig. 10(m) and (n). The local true stress of both specimens is calculated by multiplying the elastic modulus of {200} planes with its lattice strain (Fig. 10(e) and (f)), according to the Hooke's law. The elastic modulus of the {200} planes in the present material was measured as 105,772 MPa in our previous study [16]. The local true strain (ε_{xx}), obtained through DIC analysis, corresponds to the local strain at the fixed X-ray beam position. To the best of the authors' knowledge, this is the first experimental evaluation of the local strain hardening rate linked to local deformation behavior. The local strain-hardening rates of both specimens generally decreased with deformation, accompanied by periodic upwards spikes. The average local strain-hardening rates including these spikes appear to align with the strain-hardening rates observed in Fig. 5. Here, the periodic spikes over 4000 MPa correspond to serration peaks on the local true stress-strain curves, dur-

ing which the PLC band annihilates into the grip part. It is worth noting that the local strain-hardening rates of both specimens, except during the early stage of deformation ($\varepsilon \sim 0.1$), periodically dropped below the true stress-strain curves and then rose above them after some deformation time (or the true strain), with this behavior occurring more frequently in the CG specimen than in the UFG specimen. For a detailed correlation with local PLC banding, the local strain hardening rates corresponding to the DIC images labeled (1, 2, 3) for the CG specimen and (1', 2', 3') for the UFG specimen in Fig. 10(a)–(d) are indicated on their respective curves as $\square 1$, $\square 2$, $\square 3$ and $\square 1'$, $\square 2'$, $\square 3'$. Before the PLC bands reached the beam position ((1) in Fig. 10(a) and (1') in (c)), the local strain hardening rates were higher than the local true stress in both specimens ($\square 1$ in Fig. 10(m) and $\square 1'$ in Fig. 10(n)). As the PLC bands were passing through the beam position ((2) in Fig. 10(a) and (2') in Fig. 10(c)), the local strain-hardening rates dropped below the local true stress in both specimens ($\square 2$ in Fig. 10(m) and $\square 2'$ in Fig. 10(n)), indicating the onset of necking in the local area. After the PLC bands passed beyond the local area ((3) in Fig. 10(a) and (3') in Fig. 10(c)), a notable transition occurred as the local strain-hardening rates surpassed the local true stress and overcame the plastic instability condition (necking) in both specimens ($\square 3$ in Fig. 10(m) and $\square 3'$ in Fig. 10(n)). This indicates that necking was initially triggered within the propagating PLC bands, but did not progress further and was soon overcome just after the propagation of PLC bands through the high local strain hardening, which was achieved through the local multiplication of dislocations and the formation of stacking faults. Such a sequence, including temporary necking and its subsequent overcoming, was repeated throughout the entire deformation, appearing as PLC banding. Even though a greater number of dislocations are pinned by carbon atoms in the UFG specimens, the applied stress is high enough to displace (depin) the dislocations from carbons, thereby enabling their glide and triggering plastic deformation. This interpretation is supported by the observation that the UFG specimens exhibit greater plastic deformation outside the PLC band (Fig. 9), where dislocations are believed to be initially immobilized, and that greater local strain hardening is required to avoid necking and to propagate the PLC band. Indeed, it is also noteworthy that the local strain hardening rate was much higher in the UFG specimen than in the CG specimen, especially during the early stages of deformation ($\varepsilon = 0.1$ – 0.2). This high local strain hardening resulted in homogenous deformation (Figs. 3(b) and 9(c, d)), consequently leading to weak serration behavior in the UFG specimen (Fig. 2).

Direct observation of changes in the dynamic interaction between carbon atoms and mobile dislocations with grain refinement remains unclear due to experimental challenges, highlighting the need for continued investigation. Nevertheless, if macroscopic serration behavior arises from such atomistic interactions as widely recognized, this study provides insight into how these interactions could change with grain refinement and develop into characteristic localized deformation behavior. Serration behavior (or the DSA effect) is known to enhance strain-hardening ability as dislocations are repeatedly pinned by carbon atoms. However, it is also associated with localized deformation, premature failure, and surface irregularities, posing challenges for practical applications. The present study systematically examined the effect of grain refinement in high-Mn austenitic steels on macroscopic serration behavior and its correlations with local PLC banding and deformation microstructures. In this context, the present results deepen the understanding of serration behavior and local PLC banding, and offer insights into other localized deformation behavior such as Lüders deformation and necking. These findings support the development of structural metallic materials with high strength, excellent ductility, and improved surface quality.

4. Conclusions

The effect of grain refinement on the serration behavior, and further, the dynamics of Portevin-Le Chatelier banding in 22Mn-0.6C steels was thoroughly clarified with the aid of the DIC technique and *in-situ* synchrotron XRD measurement during tensile tests. The changes in local deformation behavior in the propagating PLC band with grain refinement were successfully quantified and correlated with the local strain-hardening behavior. The results are summarized as follows:

- (1) The 22Mn-0.6C steels having a wide range of the mean grain sizes, from a coarse grain of 8.4 μm to an ultrafine grain of 0.82 μm , were fabricated through repeated cold rolling and subsequent annealing process.
- (2) Serration behavior was observed on the stress-strain curves of all specimens, but the specimens with the mean grain sizes of 0.82 μm and 0.95 μm exhibited weak serration behavior during the early stage of deformation. With the aid of DIC technique applied during tensile tests, strain localization behavior characterized by the formation, propagation, and annihilation of PLC band (PLC banding) was commonly observed in all specimens. Such local PLC banding corresponded perfectly to the serration behavior on the stress-strain curves.
- (3) The PLC band velocity decreased, and the increase in local strain caused by PLC banding rose with grain refinement. That is, more deformation time and greater strain hardening were required to prevent a strain-localized region from progressing into necking and to propagate it into the adjacent area. Moreover, the strain rate within the PLC band decreased, while that beyond the band increased. During the early stage of deformation of the UFG specimen, both strain rates approached the global true strain rate, indicating that the entire gage part deformed more homogeneously and resulting in weak serration behavior on its stress-strain curve.
- (4) Through the simultaneous application of *in-situ* synchrotron XRD measurement and DIC technique during tensile tests, the crystal defects, such as dislocations and stacking faults, as well as lattice strain, significantly increased when the PLC band passed through the fixed beam position. The increase in lattice strain caused by local PLC banding was larger in the UFG specimen than in the CG specimen at an identical global tensile strain, due to the greater accumulation of the crystal defects within the propagating PLC band.
- (5) For the first time, the local strain-hardening rate was experimentally evaluated for both CG and UFG specimens in relation to local PLC banding. This evaluation revealed that necking was initially triggered within the propagating PLC bands but did not progress further, and was soon overcome by the high local strain hardening, which was achieved through the local multiplication of dislocations and the formation of stacking faults. Such a sequence, including temporary necking and its subsequent overcoming, was repeated throughout the entire deformation, appearing as PLC banding.

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.

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Sukyoung Hwang: Writing – review & editing, Writing – original draft, Visualization, Validation, Investigation, Data curation,

Conceptualization. **Myeong-heom Park:** Writing – review & editing, Validation, Supervision, Data curation. **Yu Bai:** Supervision, Methodology. **Takumi Suzumura:** Writing – review & editing, Validation, Data curation. **Ryo Asada:** Resources, Data curation. **Avala Lavakumar:** Software, Investigation, Data curation. **Jesada Punyafu:** Investigation. **Akinobu Shibata:** Validation, Supervision. **Hiroki Adachi:** Methodology. **Toshiyuki Fujii:** Writing – review & editing, Validation. **Nobuhiro Tsuji:** Writing – review & editing, Validation, Supervision, Funding acquisition, Conceptualization.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.jmst.2025.08.017](https://doi.org/10.1016/j.jmst.2025.08.017).

References

- [1] O. Grässel, L. Krüger, G. Frommeyer, L.W. Meyer, *Int. J. Plast.* 16 (2000) 1391–1409.
- [2] B.C. De Cooman, O. Kwon, K.G. Chin, *Mater. Sci. Technol.* 28 (2012) 513–527.
- [3] S. Allain, J.P. Chateau, O. Bouaziz, S. Migot, N. Guelton, *Mater. Sci. Eng. A* 387–389 (2004) 158–162.
- [4] A. Dumay, J.P. Chateau, S. Allain, S. Migot, O. Bouaziz, *Mater. Sci. Eng. A* 483–484 (2008) 184–187.
- [5] B.C. De Cooman, Y. Estrin, S.K. Kim, *Acta Mater.* 142 (2018) 283–362.
- [6] O. Bouaziz, S. Allain, C.P. Scott, P. Cugy, D. Barbier, *Curr. Opin. Solid State Mater. Sci.* 15 (2011) 141–168.
- [7] M. Koyama, in: *Encyclopedia of Materials: Metals and Alloys*, Elsevier, 2021, pp. 95–105.
- [8] S. Hwang, M. heom Park, Y. Bai, A. Shibata, W. Mao, H. Adachi, M. Sato, N. Tsuji, *Acta Mater.* 205 (2021) 116543.
- [9] J. Punyafu, S. Hwang, S. Ihara, H. Saito, N. Tsuji, M. Murayama, *Mater. Sci. Eng. A* 862 (2023).
- [10] S.J. Lee, J. Kim, S.N. Kane, B.C. De Cooman, *Acta Mater.* 59 (2011) 6809–6819.
- [11] L. Chen, H.S. Kim, S.K. Kim, B.C. De Cooman, *ISIJ Int.* 47 (2007) 1804–1812.
- [12] J.K. Kim, L. Chen, H.S. Kim, S.K. Kim, Y. Estrin, B.C. De Cooman, *Metall. Mater. Trans. A* 40 (2009) 3147–3158.
- [13] D. Liu, D. Yang, Y. Hou, Y. Li, G. Wang, H. Yi, *J. Mater. Sci. Technol.* 237 (2025) 219–255.
- [14] D. Caillard, *Acta Mater.* 112 (2016) 273–284.
- [15] A.H. Cottrell, *London, Edinburgh, Dublin Philos. Mag. J. Sci.* 44 (1953) 829–832.
- [16] S. Hwang, M.H. Park, Y. Bai, A. Lavakumar, A. Shibata, H. Adachi, M. Sato, N. Tsuji, *Scr. Mater.* 249 (2024) 116183.
- [17] Y.X. Liu, C. Hu, C.P. Huang, S. Pan, B.B. He, M.X. Huang, *Acta Mater.* 290 (2025) 120996.
- [18] Z.H. Cai, D.L. Zhang, L.F. Ma, H. Ding, Y. Feng, J. Hu, R.D.K. Misra, *Mater. Sci. Eng. A* 818 (2021) 141357.
- [19] W. Mao, S. Gao, Y. Bai, M. heom Park, A. Shibata, N. Tsuji, *J. Mater. Res. Technol.* 17 (2022) 2690–2700.
- [20] A. Alaei, H. Jafarian, A.R. Eivani, *Mater. Sci. Eng. A* 676 (2016) 342–350.
- [21] S. Niu, H. Kou, Y. Zhang, J. Wang, J. Li, *Mater. Sci. Eng. A* 702 (2017) 96–103.
- [22] J. Brechtel, S. Chen, C. Lee, Y. Shi, R. Feng, X. Xie, D. Hamblin, A.M. Coleman, B. Straka, H. Shortt, R.J. Spurling, P.K. Liaw, *Metals* 10 (2020) 1–72.
- [23] X. Gao, W. Jiang, Y. Lu, Z. Ding, J. Liu, W. Liu, G. Sha, T. Wang, T. Li, I.T.H. Chang, Y. Zhao, *J. Mater. Sci. Technol.* 154 (2023) 166–177.
- [24] K. Lu, A. Chauhan, D. Litvinov, A.S. Tirunilai, J. Freudenberger, A. Kauffmann, M. Heilmairer, J. Aktaa, *J. Mater. Sci. Technol.* 100 (2022) 237–245.
- [25] Z.C. Luo, M.X. Huang, *Scr. Mater.* 142 (2018) 28–31.
- [26] Y.N. Dastur, W.C. Leslie, *Metall. Mater. Trans. A* 12 (1981) 749–759.
- [27] K. Renard, S. Ryelandt, P.J. Jacques, *Mater. Sci. Eng. A* 527 (2010) 2969–2977.
- [28] A. Saeed-Akbari, A.K. Mishra, J. Mayer, W. Bleck, *Metall. Mater. Trans. A* 43 (2012) 1705–1723.
- [29] M. Koyama, E. Akiyama, K. Tsuzaki, *J. Iron Steel Inst. Jpn.* 100 (2014) 1123–1131.
- [30] S.Y. Lee, S.I. Lee, B. Hwang, *Mater. Sci. Eng. A* 711 (2018) 22–28.

- [31] M. Koyama, T. Sawaguchi, K. Tsuzaki, *ISIJ Int.* 55 (2015) 1754–1761.
- [32] S.J. Lee, J. Han, S. Lee, S.H. Kang, S.M. Lee, Y.K. Lee, *Sci. Rep.* 7 (2017).
- [33] W. Charnock, *Philos. Mag.* 18 (1968) 89–99.
- [34] P.G. McCormick, *Philos. Mag.* 23 (1971) 949–956.
- [35] Y. Saito, N. Tsuji, H. Utsunomiya, T. Sakai, R.G. Hong, *Scr. Mater.* 39 (1998) 1221–1227.
- [36] Y. Saito, H. Utsunomiya, N. Tsuji, T. Sakai, *Acta Mater.* 47 (1999) 579–583.
- [37] S. Hwang, Y. Bai, S. Gao, M.H. Park, A. Shibata, N. Tsuji, *Mater. Sci. Eng. A* 894 (2024) 146193.
- [38] S. Hwang, H. Kato, K. Okada, M. heom Park, A. Lavakumar, R. Gholizadeh, H. Adachi, M. Sato, N. Tsuji, *Mater. Res. Lett.* 12 (2024) 571–579.
- [39] Y.Z. Tian, Y. Bai, M.C. Chen, A. Shibata, D. Terada, N. Tsuji, *Metall. Mater. Trans. A* 45 (2014) 5300–5304.
- [40] N. Tsuji, S. Ogata, H. Inui, I. Tanaka, K. Kishida, S. Gao, W. Mao, Y. Bai, R. Zheng, J.P. Du, *Scr. Mater.* 181 (2020) 35–42.
- [41] M.R. Daymond, M.A.M. Bourke, R.B. Von Dreele, B. Clausen, T. Lorentzen, *J. Appl. Phys.* 82 (1997) 1554–1562.
- [42] L. Balogh, G. Ribárik, T. Ungár, *J. Appl. Phys.* 100 (2006).
- [43] M.S. Paterson, *J. Appl. Phys.* 23 (1952) 805–811.
- [44] B.E. Warren, *Prog. Met. Phys.* 8 (1959) 147–202.
- [45] W. Mao, S. Gao, W. Gong, T. Kawasaki, T. Ito, S. Harjo, N. Tsuji, *Acta Mater.* 278 (2024) 120233.
- [46] W. Woo, J.S. Jeong, D.K. Kim, C.M. Lee, S.H. Choi, J.Y. Suh, S.Y. Lee, S. Harjo, T. Kawasaki, *Sci. Rep.* 10 (2020) 1350.
- [47] R.W. Cheary, A.A. Coelho, J.P. Cline, *J. Res. Natl. Inst. Stand. Technol.* 109 (2004) 1–25.