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ORIGINAL REPORT



In situ high-energy X-ray diffraction mapping of Lüders band propagation in medium-Mn transformation-induced plasticity steels

Minghe Zhang^a, Runguang Li^a, Jie Ding^a, Haiyang Chen^a, Jun-Sang Park^b, Jonathan Almer^b and Yan-Dong Wang^a

^aBeijing Advanced Innovation Center for Materials Genome Engineering, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, People's Republic of China; ^bX-ray Science Division, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

ABSTRACT

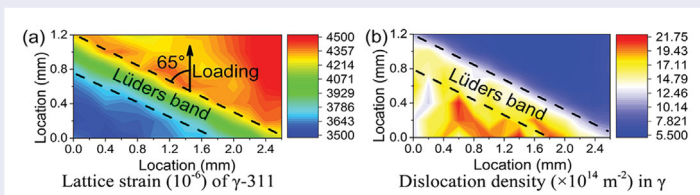
Two-dimensional distributions of microstructural characteristics of austenite (γ) around the Lüders band in medium-Mn transformation-induced plasticity (TRIP) steel were revealed by in situ high-energy X-ray diffraction. Lüders band propagation leads to significant changes in the volume fraction and lattice strains of austenite. The evolution of the lattice strain of γ -311 accords well with the true yield behavior model and is used to estimate the angle of the Lüders band front with respect to the tensile axis. The modified Williamson-Hall analysis shows that the dislocation density in austenite increased from $7 \times 10^{14} \text{ m}^{-2}$ to $1.5 \times 10^{15} \text{ m}^{-2}$ after Lüders band propagation.

ARTICLE HISTORY

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KEYWORDS

Lüders band; medium-Mn TRIP steel; high-energy X-ray diffraction



IMPACT STATEMENT

This in situ high-energy X-ray diffraction study unravels two-dimensional distribution of microstructural evolution during Lüders band propagation in medium-Mn TRIP steels.

1. Introduction

Due to their excellent strength-ductility combination and reasonable production costs, medium-Mn transformation-induced plasticity (TRIP) steels have received considerable attention for potential applications in modern automobiles [1–4]. The amount and stability of retained austenite (RA) as well as developing the mechanical properties as a function of alloying contents and intercritical annealing conditions have been systematically investigated in previous studies [5–10]. It was shown that both the strength and ductility of medium-Mn TRIP steels were significantly enhanced with cold-rolled and intercritical annealing in comparison with hot-rolled and intercritical annealing [7,8]. However, extensive Lüders band propagation was reported for medium-Mn TRIP steels during tensile deformation after cold rolling and

intercritical annealing [9–12], which is undesirable on the surface of the sheet during forming.

The Lüders band is a plastic instability phenomenon that corresponds to a stress plateau stage in the stress-strain curve, which often occurs during the tensile deformation of metallic materials [12]. A classic theory about this phenomenon is that the solute interstitials such as C and N atoms interact with dislocations, forming a Cottrell atmosphere [13]. The digital image correlation (DIC) method and infrared thermography (IRT) technique are used to study the kinematic and thermal characteristics of Lüders band propagation [12,14]. Transmission electron microscopy (TEM) experiments can only characterize the microstructures of thin-foil samples, which cannot truly reveal the change in the stress/strain state and microstructural features in bulk materials during Lüders

CONTACT Yan-Dong Wang ✉ ydwang@mail.neu.edu.cn Beijing Advanced Innovation Center for Materials Genome Engineering, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, People's Republic of China

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band propagation [15]. The in situ high-energy X-ray diffraction (HE-XRD) technique can not only reveal the detailed scenario on phase transformation but also investigate the dislocation evolution of constituent phases during deformation [6,16]. In the present study, we used the in situ HE-XRD technique to map the distributions of the phase volume fraction, lattice strain and dislocation density around the Lüders band, unraveling the complexities of Lüders band propagation.

2. Experiment

A medium-Mn steel with a chemical composition of Fe-0.12C-10.16Mn-1.87Al (in mass%) was produced through a vacuum furnace. After being homogenized at 1200°C for 1 h, plates with 20 mm thickness were further hot-rolled between 900°C and 1100°C to a thickness of 4 mm and then cold-rolled to a thickness of 1.5 mm. Specimens for microstructural observations and tensile tests were cut from thin plates by electrosparking. Subsequently, the specimens were annealed at 675°C for 1 h and then air-cooled. The microstructural observation was performed using a ZEISS SUPRA55 field-emission scanning electronic microscope (FE-SEM) after etching with 4 vol.% Nital. The quantitative analysis of the grain sizes of ferrite and RA were conducted using Image-Pro Plus 6.0 (produced by Media Cybernetics company, USA) image analysis software using an SEM micrograph [4]. The volume fraction of RA was determined by using the method described in [17].

The in situ HE-XRD experiments were performed at the 1-ID-E endstation of the Advanced Photon Source (APS) at Argonne National Laboratory. A schematic drawing of the experimental setup is shown in Figure 1. A monochromatic X-ray beam with an energy of 71.676 keV (wavelength 0.017297 nm) was used. The beam was slitted to 0.2 mm × 0.2 mm. The tensile tests were

performed at room temperature with a nominal strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ using a servo-hydraulic MTS load frame under the displacement control mode. The dog-bone-shaped tensile specimen with the gauge part of 10 mm (length) × 3 mm (width) × 0.5 mm (thickness) was mounted with the rolling direction parallel to the loading direction (LD). During deformation, the HE-XRD data was collected using 4 GE detectors placed 1770 mm from the sample. The two-dimensional diffraction data was transformed to a one-dimensional line profile by integrating over a $\pm 5^\circ$ azimuth range around the LD of the specimen. The diffraction peaks were fit to pseudo-Voigt functions using MATLAB programs developed by the Materials Physics and Engineering group at the APS. Here, {200}, {220}, {311}, {420}, {422} and {511} diffraction peaks of austenite were mainly focused to elucidate changes in the elastic strain/stress field and the dislocation density of austenite during Lüders band propagation. Using the peak position information, lattice strains were obtained by calculating the change in lattice spacing relative to the lattice spacing in the undeformed state. The peak width information measured by the full width at half-maximum (FWHM) was used to estimate the evolution of dislocation density of austenite around the Lüders band. The instrumental broadening was characterized using NIST CeO₂ powder and removed from the total peak width measured during in situ experiments. To characterize a region around the Lüders band, the loading was paused once the Lüders band was detected and the sample was held at a particular displacement. An area around the Lüders band was scanned by moving the sample by 1.2 mm and 2.6 mm in the directions parallel and perpendicular to the LD, respectively. The step size of the sample movement was 0.2 mm along both directions. After the two-dimensional scanning was completed, the loading was continued until sample failure.

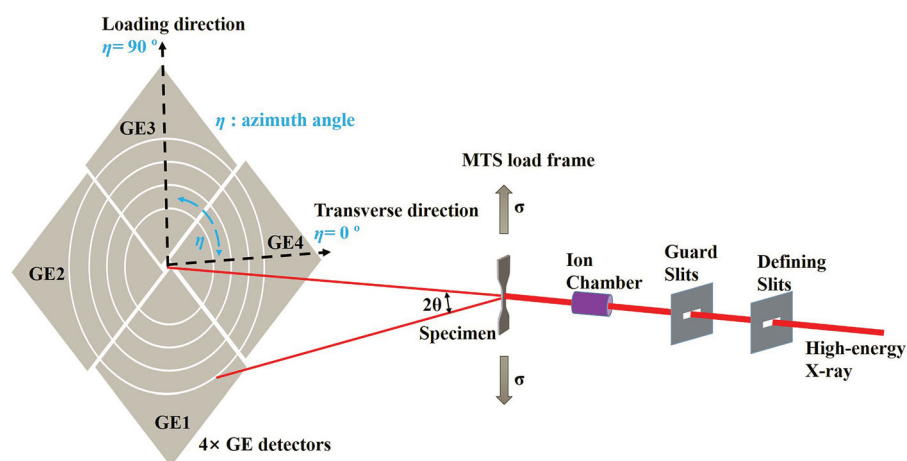


Figure 1. Experimental setup of the tensile test with in situ HE-XRD experiments.

3. Results and discussion

Figure 2(a) shows the annealed microstructure, which consists of fine grains of austenite (γ) and ferrite (α). The grain sizes of the austenite and ferrite are approximately $0.78\ \mu\text{m}$ and $0.47\ \mu\text{m}$, respectively. Figure 2(b) shows the in situ stress-strain curve overlapped with the evolution of RA as a function of the engineering strain. The yield strength (YS) and ultimate tensile strength (UTS) of the steel investigated in this work are 820 MPa and 1330 MPa, respectively. The Lüders strain is 7% and the total elongation (TE) is 31%. The fluctuation of stress during Lüders straining is due to a pause in loading (approximately 30 minutes) when X-ray scanning measurements were performed. Before loading, the initial volume fraction of RA in the experimental steel was 81%. The volume fraction of austenite suddenly decreased from 81% to 52% at an applied strain of 0.04 (at the middle of Lüders straining), which indicates that martensitic transformation of studied steel during tensile deformation is strain-induced. After Lüders band propagated the entire gauge of the sample, austenite transformed to α' -martensite gradually until sample failure.

Figure 3(a) shows the map of volume fraction (%) of γ , showing a high volume fraction of γ approximately 81%

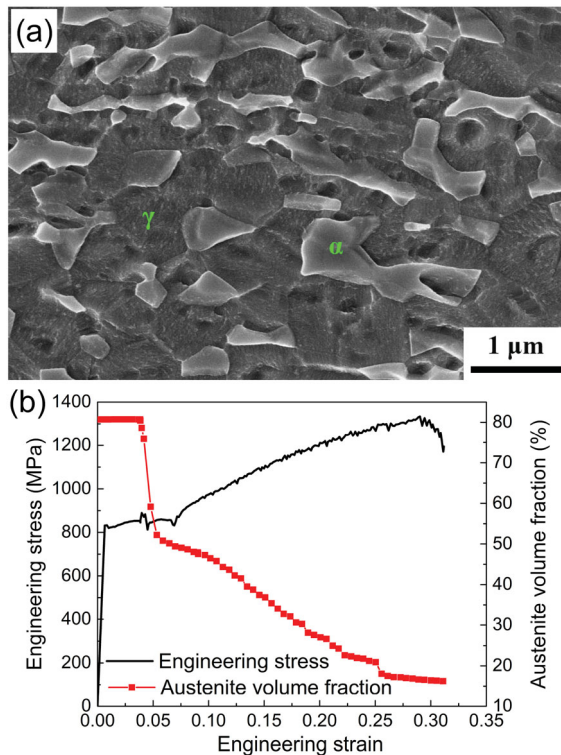


Figure 2. (a) An SEM micrograph of the experimental steel; (b) Engineering stress-strain curve and change in austenite volume fraction of medium-Mn TRIP steels during in situ loading for HE-XRD experiments.

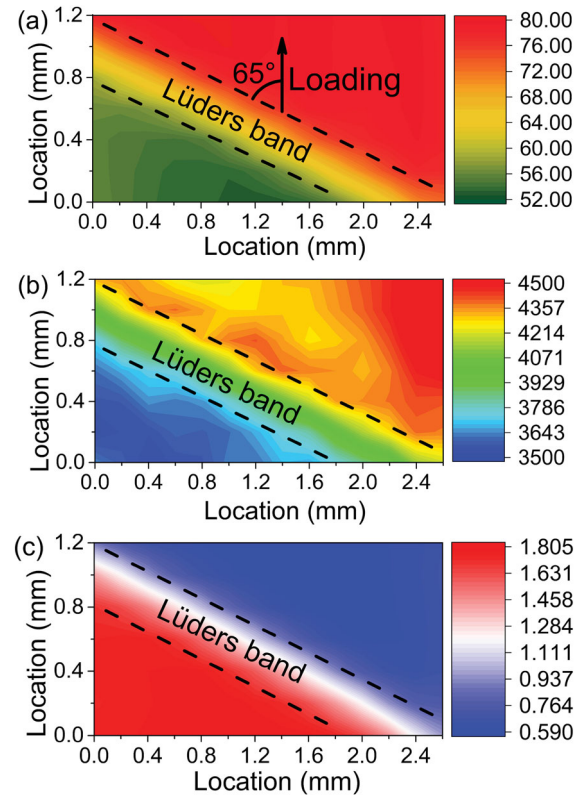


Figure 3. (a) Map of volume fraction (%) of γ ; (b) Map of lattice strain (10^{-6}) of γ -311; (c) Map of FWHM (10^{-3} rad) of γ -311. The areas between two dash lines correspond to the Lüders band and the arrow demonstrates the loading direction (in the same direction as Lüders band propagation).

in front of the Lüders band and a low volume fraction of γ approximately 52% behind Lüders band located in the higher right portion and lower left portion of the map, respectively. Lüders band propagation led to microstructures behind the Lüders band located in the lower left portion of the maps, consisting of 52% γ , 19% α and 29% α' . Figure 3(b) shows the map of the lattice strains of γ -311. It can be seen that γ -311 exhibited a higher lattice strain in front of the Lüders band and a lower lattice strain after the Lüders band propagation. Once the Lüders band propagated through the certain gauge of the sample, there was a decrease of $800\ \mu\epsilon$ (where $1\ \mu\epsilon$ represents a strain of 10^{-6}) for the γ -311 lattice strain. The sudden decrease in the lattice strain during Lüders band propagation could be due to two factors, i.e. the volume expansion from γ -to- α' transformation and the loading partitioning among constituent phases [6]. Figure 3(c) shows the map of FWHM for the γ -311, showing that Lüders band propagation leads to a large increase in FWHM for γ -311.

The inclination angle φ of the Lüders band front with respect to the tensile axis is 65° , which can be seen clearly from the maps of volume fraction, lattice strains and

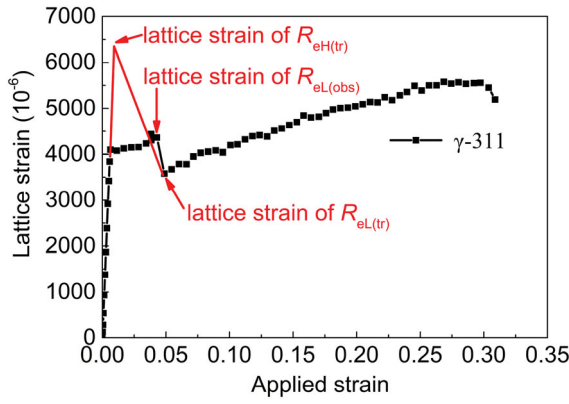


Figure 4. The evolution of the γ -311 lattice strain as a function of the applied strain.

FWHM of γ in Figure 3. The angles φ may vary from 45° to 70° in steels, depending on the microstructures [18,19]. An essential point about the formation of such angles is that the plastic deformation observed near well-developed Lüders fronts is not pure shear but a combination of shear strain and a principal strain perpendicular to the Lüders fronts [20]. According to the model by Schwab and Ruff [20], the true upper yield strength ($R_{eH(tr)}$) is much higher than the observed yield strength ($R_{eL(ops)}$) and the true material behavior after first yielding exhibits normal strain hardening starting at the true lower yield strength ($R_{eL(tr)}$). Our experimental finding on the evolution of the γ -311 lattice strain as a function of the applied strain, as shown in Figure 4, agrees well with the abovementioned model. Due to Lüders band propagation, the lattice strain of γ -311 decreased from $4400 \mu\epsilon$ to $3600 \mu\epsilon$, which could be considered the axial strain ϵ_{11} of $R_{eL(ops)}$ and $R_{eL(tr)}$ of austenite, respectively. And the corresponding transverse strain ϵ_{22} of $R_{eL(ops)}$ and $R_{eL(tr)}$ of austenite are $-1900 \mu\epsilon$ and $-1700 \mu\epsilon$, respectively. While different approaches [21–23] can be used to obtain the stress (and the effective stress) in a polycrystalline material using the lattice strain data, the effective stress of austenite was obtained using the approach outlined in Ref. [23]. The elastic modulus E_{311} and Poisson's ratio ν_{311} were 190 GPa and 0.28, respectively, which were obtained from plots of the applied stress vs. lattice strains during the elastic stage. Thus, the $R_{eL(ops)}$ and $R_{eL(tr)}$ of austenite were estimated to be 935 MPa and 787 MPa, respectively. The relationship among $R_{eL(ops)}$, $R_{eH(tr)}$ and $R_{eL(tr)}$ can be expressed as:

$$R_{eL(ops)} = \frac{R_{eH(tr)} + \sqrt{R_{eH(tr)}^2 + 8R_{eL(tr)}^2}}{4} \quad (1)$$

Therefore, the true higher yield strength of austenite is calculated to be 1208 MPa. As depicted in Ref. [20], the angel φ is only a function of $R_{eH(tr)}$ and $R_{eL(tr)}$ and is

expressed as:

$$\phi = \frac{\pi}{2} - \frac{1}{2} \arctan \left(2 \sqrt{\frac{4R_{eL(tr)}^2 - R_{eH(tr)}^2 + R_{eH(tr)}}{\sqrt{R_{eH(tr)}^2 + 8R_{eL(tr)}^2}}} \right) \quad (2)$$

The angle φ is calculated to be 60° , which is approximately equal to the observed 65° .

Generally, the FWHM values increase with the applied strain, indicating a high level of subgrain-scale activities during plastic deformation [24]. The FWHM of face-centred-cubic (FCC) γ peaks were evaluated by modified Williamson-Hall (W-H) plot. The fundamental equation for the modified W-H plot is:

$$\Delta K \cong 0.9/D + \sqrt{\frac{\pi M^2 b^2}{2}} \sqrt{\rho} (KC^{0.5}) \quad (3)$$

where $K = 2\sin\theta/\lambda$; $\Delta K = 2\cos\theta(\Delta\theta)/\lambda$; θ and λ are the diffraction angle and the wavelength of X-rays, respectively; D , ρ and b are the average grain or particle size, dislocation density and the Burgers vector of dislocations, respectively; M is a constant depending on the effective outer cut-off radius of dislocations; and C is the dislocation contrast factor. In this study, $M = 2$ was used to give the level of accuracy expected by the modified W-H analysis. Values for C_{hkl} for edge type $\{111\} <110>$ and screw type $\{111\}$ dislocations in γ can be calculated using the ANIZC software [25], with elastic constants inputs of $c_{11} = 164$ GPa, $c_{12} = 113$ GPa, $c_{44} = 133$ GPa [26]. To find the optimum C_{hkl} , the modified W-H fitting using the C_{hkl} with the screw/edge ratio ranging from 0 to 1 was performed with a 0.01 step size, and the C_{hkl} that gave the highest R -square value was selected [24]. The analysis of the modified W-H plots of the two selected points located in front of and behind the Lüders band are shown in Figure 5(a) and Figure 5(b), respectively, and the location of the two points are marked in Figure 5(c). The slope obtained by modified W-H plots is proportional to the square root of the dislocation density ρ , and the intercept is inversely proportional to the average grain size D . We estimated that the dislocation density and grain size of the austenite in front of the Lüders band are $7.06 \times 10^{14} \text{ m}^{-2}$ and $5.7 \mu\text{m}$, respectively. Such dislocation density is due to the combination of cold rolling and intercritical annealing. The estimated grain size of austenite in front of the Lüders band may not be statistically accurate because the grain size of the austenite is in the submicrometre range for the studied steel. For the selected point behind the Lüders band, the estimated dislocation density and grain size of austenite

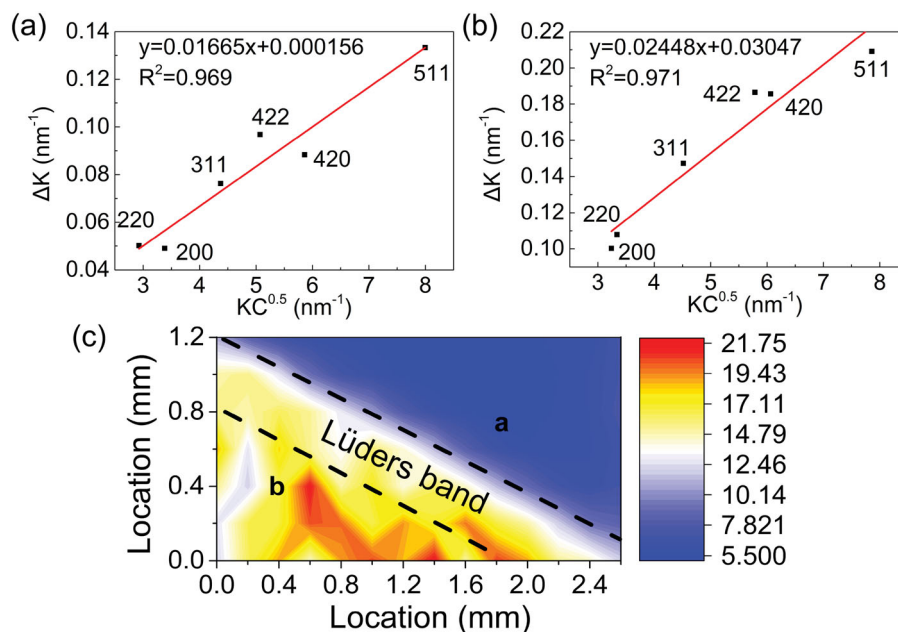


Figure 5. (a) The modified W–H plot in front of the Lüders band; (b) The modified W–H plot behind the Lüders band; (c) Map of dislocation density ($\times 10^{14} \text{ m}^{-2}$) in austenite of experimental steel.

are $1.52 \times 10^{15} \text{ m}^{-2}$ and 29 nm, respectively. This finding indicates the austenite phase after Lüders band propagation contains one dislocation within each grain on average. The distribution of the dislocation density in austenite is shown in Figure 5(c). It can be seen that the dislocation density of austenite in front of the Lüders band is approximately $7 \times 10^{14} \text{ m}^{-2}$ while the dislocation density of austenite behind the Lüders band is higher than $1.5 \times 10^{15} \text{ m}^{-2}$ on average. The significant increase in the dislocation density of austenite results from a high volume fraction of the phase transformation from austenite to martensite inducing a large volume expansion during Lüders band propagation.

The Lüders bands correspond to a stress plateau stage in the stress-strain curve with almost zero strain hardening, which is associated with a localized deformation in the banded structures. Usually, a very low strain hardening rate or large strain localization may result in samples failure during plastic deformation [27]. In contrast, many metallic materials undergo Lüders band propagation without sample failure [18,28,29]. In our study, by combining Figures 3 and 5, we can see that the sharp transitions in volume fraction, lattice strain and dislocation density of austenite are on a length scale approximately 0.4 mm. This outcome indicates that there are large local gradients of strain/stress and microstructures around the Lüders band. Such gradients could provide effective localized work-hardening capability and contribute to the stability of Lüders band propagation through the entire specimen without failure. The large and complex change in the lattice strain and the distribution of

dislocation density in austenite around the Lüders band unraveled by in situ HE-XRD technique not only further extend the knowledge related to medium-Mn TRIP steels but also promise to deepen the understanding of Lüders deformation in other alloy systems.

4. Conclusion

In summary, our in situ HE-XRD experiments studied the two-dimensional distributions of the microstructural characteristics of austenite during Lüders band propagation in medium-Mn TRIP steels. The results revealed that Lüders band propagation not only promotes high volume fraction of austenite transformation to martensite but also leads to large and complex changes in the lattice strains in austenite. Based on the true yield behavior model, the calculated angle (60°) of the Lüders band front with respect to the tensile axis is approximately equal to the observed one (65°). The increase in the dislocation density in austenite from about $7 \times 10^{14} \text{ m}^{-2}$ to higher than $1.5 \times 10^{15} \text{ m}^{-2}$ caused by Lüders band propagation was quantified.

Disclosure statement

No potential conflict of interest was reported by the authors.

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ORCID

Runguang Li  <http://orcid.org/0000-0001-5395-0470>

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