Heterogeneous Distribution of Microstrain Evolved During Tensile Deformation of Polycrystalline Plain Low Carbon Steel

Hai Qiu *, Rintaro Ueji, Yuuji Kimura and Tadanobu Inoue

Research Center for Structural Materials, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan; Ueji.Rintaro@nims.go.jp (R.U.); Kimura.Yuuji@nims.go.jp (Y.K.); Inoue.Tadanobu@nims.go.jp (T.I.)

* Correspondence: QIU.Hai@nims.go.jp; Tel.: +81-029-859-2107

Received: 18 April 2020; Accepted: 9 June 2020; Published: 10 June 2020

Abstract: On a macroscale, the stress–strain curve of polycrystalline steel exhibits perfectly linear behavior in the elastic tension region. We observed that the strain distribution within grains is inhomogeneous in the range of the elastic deformation region. Microstrain concentrates at some local sites which are at and near the grain boundaries or in the interior of the grains. The microstrain is pseudo-periodic, and its period increases with applied stress, tending to approach the grain size. In addition to the tension-strain concentration, the compression-strain concentration is even present in the elastic region. The pseudo-periodic and inhomogeneous microstrain is attributed to the orientation heterogeneity of grains in polycrystalline steel.

Keywords: microstrain; polycrystalline steel; tensile test; grain; ferrite; digital image correlation

1. Introduction

In a polycrystalline steel, grain boundary has significant influence on its mechanical properties. There are many studies [1–6] clarifying the mechanism by which the grain boundary affects plastic deformation, and these studies have been conducted by various methods. On a microscopic scale, elastic incompatibility occurs due to different crystal orientations, resulting in heterogeneous deformation at grain boundaries [1,2]. Finite element method (FEM) calculations have also showed the possibility of strain localization in polycrystals [3]. Hook and Hirth designed a sample with bicrystals of a high purity Fe-3% Si alloy [4]. They investigated the dislocation slip occurring in the bicrystals subjected to small plastic strains by experiment and verified that heterogeneous deformation occurred at grain boundaries. Raabe et al. found that grain-to-grain plastic strain heterogeneity and in-grain plastic strain heterogeneity occurred in large macroscopic plastic strains [5,6].

Occasionally, heterogeneous deformation links to microyielding, which occurs at local sites in polycrystalline metals subjected to applied stresses even smaller than the yield stress. For example, in silicon–iron, microyielding begins to take place in a grain at a stress level of 79 percent of the lower yield stress, and the number of microyielded grains increases as applied stress increases [7]. Around 70%–80% of grains (in volume fraction) are microyielded before macroscopic yielding begins to occur in the interstitial free (IF) steel [8].

The mechanism of microyielding has been examined by transmission electron microscope (TEM), and evidences of dislocation emission from grain boundaries and dislocation pile-up were found [9,10]. Dislocation pile-up contributes to yielding [9,10]. Although TEM observation revealed the nature of microyielding, due to its extremely small observation area, it cannot capture the whole image of microyielding in a grain. Therefore, to investigate the microyielding distribution within a grain,
observation should be performed on a relatively large scale—for example, grain-scale. Twenty years ago, there was no reliable method measuring microstrain within grains. Hence, slip line \cite{8,11} or etch pit \cite{7,12} was used to identify microyielding. This method provides evidence directly linking dislocation motion with crystallography, but it is difficult to conduct the quantitative evaluation of microstrain corresponding to the microyielding. Consequently, if microstrain distribution is evaluated before and around the yielding, it will help us to understand the microyielding. However, microdeformation behavior on grain-scale in a polycrystal at a small strain, especially in the elastic region, has been studied less.

In this study, the aim is to investigate microstrains in polycrystalline steel with the focus on a grain-scale in the macroscopic elastic region. Digital image correlation (DIC) is a technique for measuring displacement and strain field on the surface of an object \cite{5,6,13–18}. In recent years, small-scale DIC has been developed to measure the local deformation response of materials at micro-scale and meso-scale \cite{19–22}. Subset is the unit in operating the DIC. Each subset is uniquely identified by its gray-scale intensity, and thus, it is required for the specimen to be properly speckled with a high-contrast, random and dense speckle pattern \cite{19}. The quality and density of a speckle pattern is probably the most important factor. For different length-scales, different speckles should be used \cite{20}. Kammers et al. \cite{21} provided a thorough survey of small-scale patterning methods. In addition, at microstructural scale, inherent noise of the DIC measurement is also another important factor affecting the accuracy of the DIC measurement \cite{19,22}. Ravindran et al. \cite{19,22} found that an accurate selection of DIC parameters can ensure high strain-to-noise ratios, reducing the effect of noise.

By using the DIC technique, a tensile test on a ferrite steel in which the applied stress was below the upper yield point was observed in situ and the microstrain distribution within grains was studied.

2. Materials and Methods

A steel bar (14 mm × 14 mm × 55 mm) with 0.05% C, 0.01% Mn, <0.008% Si, <0.002% P, 0.002% S, 0.002% Al, 0.006% Cr, <0.002% Mo, 0.002% V, <0.01% Ni, <0.001% Cu, <0.001% Sn, 0.0027% N, 0.0032% O and the balance Fe (in wt%) was heat treated by three processes as follows: 1 heating it up to 900 °C, keeping for 5 min at 900 °C, and then air cooling down to room temperature; 2 repeating process 1 again; 3 heating the sample up to 900 °C, keeping for 5 min at 900 °C, followed by furnace cooling down to room temperature. Due to relatively clean chemical composition, segregation can be neglected. A plate tensile specimen with a parallel part 14 mm long, 3 mm wide, and 0.32 mm thick was machined from the steel bar (see Figure 1a). The front surface of the specimen was polished, followed by etching with 1.5% nital. Its microstructure on the front surface was observed by optical microscope and electron back scattered diffraction (EBSD). The EBSD measurement was performed on an area of 2 mm × 0.8 mm in the center of the specimen with step 2 µm. Afterwards, random speckle was prepared on the front surface and a strain gauge with an area of 1 mm (length) × 1.1 mm (width) was attached to the back front (see Figure 1b). The speckle was prepared as follows: 1 placing black toner on the surface of the specimen; 2 blowing out the toner with compressed air (toner cluster was blown off, and only fine toner particles were left); 3 repeating process 1 and 2 several times to obtain optimal particle density; 4 covering a layer of silver (thickness 1.8 nm).

The specimen was tensioned at a crosshead speed of 0.1 mm/min at room temperature with the experimental setup shown in Figure 2. The deformation process of the front surface within the macroscopic elastic region (from zero to the upper yield point) was recorded using a CCD camera (Grasshopper GRAS-505SM/C, 2448 pixel × 2048 pixel). The obtained image size is 0.41 mm × 0.34 mm. DIC operation was performed on the digital images taken in the tension process by software of VIC-2D (Correlated Solutions, Inc.) (subset size: 9 pixel × 9 pixel (4.8 µm × 4.8 µm); step: 5 pixel (1.7 µm)) to determine the displacement and microstrain within grains. In the DIC operation, the displacement uncertainty is 0.02 pixel. The strain obtained from the strain gauge is taken as the macrostrain of the tensile specimen.
3. Results

3.1. Microstructure

The optical microstructure of the steel is shown in Figure 3a. It is composed of ferrite and pearlite (volume fraction 0.8%). It can be seen that the ferrite has equi-axed grains and the average grain size is 30.4 µm. The crystal orientation was measured by using EBSD and the inverse pole figure (IPF) of the steel is shown in Figure 3b. It is noted that the DIC measurement region is within the EBSD measurement region—and to match the DIC measurement region—Figure 3b was cropped from the whole IPF map whose size is 2 mm × 0.8 mm. Figure 3b shows that the crystal orientation is random. These experimental results indicate that the applied steel is an approximately ideal model material of bcc (body-centered cubic) structure.

3.2. Noise level of the DIC Strain Measurement

DIC strain measurement has its inherent noise, which is dependent on the experimental setup, speckle and DIC parameters [19,22]. Since the aim of the present study is to measure the microstrain heterogeneity at grain scale, to allow for high spatial resolution of local information, we selected the subset size as small as possible. The subset size was 9 pixel × 9 pixel and step size of 5 pixels. A set of four images of the unloaded specimen was captured to determine the average DIC strain noise level which was evaluated in terms of strain noise and bias level. Ravindran et al. [19,22] suggested to use...
standard deviation (STD) and average strain to evaluate the strain noise and bias level, respectively. The two parameters were obtained by correlating the four images with the same subset size (9 pixel × 9 pixel) and step size (5 pixel), and their evolution with filter size is shown in Figure 4a. Large filter size decreases the noise and bias levels. However, using large subset will smear out the local information, contradicting the aim of the present study. An appropriate filter size, 15, was selected on the balance of spatial resolution and noise level, at which the bias level is only about 8 µε and the STD is relatively low. This bias level can be neglected.

Noise-to-strain ratio, standard deviation (STD) divided by the global strain, was proposed to reflect the effect of DIC strain measurement noise on the strain measurement [19,22]. The noise-to-strain ratio in the tensile process was calculated with the STD at filter 15, and the result is shown in Figure 4b. The effect of noise decreases with the applied strain (far-field strain). If the noise and real strain levels have almost the same order, the strain uncertainty is great. We take 0.5 as an acceptable noise-to-strain ratio, below which the effect of noise is regarded to be small. This value corresponds the far-field strain level of about 180 µε, i.e., the applied stress level of about 38 MPa.

3.3. Microstrain within Grains

The global stress–strain curve is shown in Figure 5a. The stress linearly increases with macrostrain below 130 MPa; the stress–strain curve then gradually deviates from the straight line. The linear
relation between the stress and strain indicates that the bulk steel exhibits elastic behavior below 130 MPa. When the stress continuously rises from 130 MPa to the upper yield stress (320 MPa), macroscopic plasticity begins to occur and gradually increases, although it is still in the so-called macroscopic elastic region. After the upper yield stress, macroscopic yielding takes place, followed by Lüders yield plateau. It is noted that the limit of the elastic region (130 MPa) in Figure 5 is determined from the stress–strain curve obtained from the strain gauge attached to the back surface.

DIC analysis of the digital images gives the microstrain field in terms of the strain contour. Figure 6 shows the two-dimensional strain contours ($\varepsilon_x$ field) at the applied stresses of 26 MPa, 128 MPa and 307 MPa. The two-dimensional strain contours indicate that the microstrain is inhomogeneous in the macroscopic elastic region. The microstrain localizes at some local sites. Some of the local sites are on the grain boundaries, and some are in the interior of grains. It is known from Figure 4b that the macrostrain at 26 MPa (Figure 6a) has almost the same order as the DIC measurement noise. As a result, the effect of noise is great in Figure 6a.

![Figure 5](image)

**Figure 5.** (a) Global stress–strain curve; (b) stress–strain curve in the macroscopic elastic region. Solid curve was measured by strain gage. ○ represents the average strain of the two-dimensional strain contours obtained by DIC.

The average value of the two-dimensional strain contour, i.e., the average microstrain is plotted in Figure 5b. ○ in Figure 5b represents this average microstrain. The average microstrain agrees with the macrostrain below a stress level of 130 MPa; beyond the elastic region, the average microstrain is larger than the macrostrain for a given stress level. The deviation between the microstrain and macrostrain is attributed to the significant strain concentration on a grain scale.

![Figure 6](image)

**Figure 6.** Two-dimensional strain contours with grain boundaries at the applied stresses of (a) 26 MPa, (b) 128 MPa and (c) 307 MPa.

Grain-to-grain and in-grain microstrain heterogeneity has intuitively been described by the two-dimensional strain contour. Further quantitative evaluation is performed by one-dimensional analysis. A line (line AB) is drawn in Figure 3b. It is parallel to the tension direction (x axis). The strain
(\(\varepsilon_x\)) along this line at different stress levels is extracted from the two-dimensional strain contours and is shown in Figure 7a–c. It is noted that the contours in Figure 7a–c are \(\varepsilon_x\) contours with grain boundaries around line AB. Macrostrain derived from the strain gauge is illustrated by the red line in Figure 7, as a baseline. If the microstrain distribution is uniform, the microstrain anywhere should be equal to the macrostrain, i.e., the microstrain distribution should be a horizontal line. However, the microstrain goes up and down the baseline, and its distribution has periodicity. Its period reflects the density of strain localization, and its amplitude indicates the degree of strain concentration. With the increase of the applied stress, the microstrain wave becomes rougher. At an applied stress of 307 MPa (Figure 7c), some local places (red regions) show significantly stronger concentration than the surrounding regions. Their local strain is larger than 5000 \(\mu\varepsilon\), inevitably higher than the critical strain for yielding. These places are only in some local regions, not over the whole grain. This means that these local sites are in the initial state of microyielding and the microyielding does not spread over the whole grain.

**Figure 7.** Microstrain distributions at different applied stresses along line AB. (a) applied stress 26 MPa and macrostrain 119 \(\mu\varepsilon\); (b) applied stress 128 MPa and macrostrain 513 \(\mu\varepsilon\); (c) applied stress 307 MPa and macrostrain 1341 \(\mu\varepsilon\). x direction is the tension direction.
In order to find out the cause for this microyielding, the characterization of the microstructure around the highest microstrain peak was examined. The point-to-point misorientation angle along line AB was calculated from the EBSD data and is shown in Figure 8a. The local elastic modulus along line AB was also calculated with the crystallographic orientation parallel to the loading direction and the elastic stiffnesses of pure iron single crystal ($c_{11} = 231.4$ GPa, $c_{12} = 134.7$ GPa, $c_{44} = 116.4$ GPa) [23]. Figure 8b shows the local elastic modulus distribution. A large microyielding site (concentrated large deformation) at grain boundary is indicated by an arrow. $\Delta \theta$ and $\Delta E$ represent the differences in the misorientation angle and local elastic modulus between the right grain and left grain, respectively. Both of them are great. It seems that they are the crucial factors affecting the microstrain concentration. However, the left neighboring boundary having almost the same order of $\Delta \theta$ and $\Delta E$ did not induce microstrain concentration (see Figure 7c). Great $\Delta \theta$ and $\Delta E$ indicate the capacity inducing great microstrain concentration, but Figure 8 shows that this capacity does not inevitably forms microyielding at grain boundary. It is believed that constraint from the neighboring grains can result in a significantly complex state of deformation on a specific grain [22,24,25]. Such a specific deformation state probably results in negligible incompatibility of elastic deformation, even if adjacent grains have great misorientation angle and significant difference in the elastic modulus. Strain-free grains are present in AISI 1018 steel even at the applied strain level of 4.4% [22].

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Figure 8. Distribution of (a) misorientation angle and (b) local elastic modulus along line AB. Dotted lines indicate the positions of grain boundaries.

As shown in Figure 7, microstrain peaks are above or below the baseline. Strain localization greater than the baseline was defined as positive localization and strain localization less than the
baseline as negative localization. Nine horizontal lines were drawn to uniformly divide the whole area of the two-dimensional strain contour shown in Figure 6. Line AB in Figure 3b is one of nine horizontal lines. The period and amplitude (peak value) of the microstrain distribution along each line at different stress levels were summarized, and the average values of the nine lines are given in Figure 9. Figure 9a shows the period which enlarges with the applied stress and gradually approaches to the grain size. This indicates that the number of strain localization decreases with increasing macrostrain. It is known that plastic deformation produces substructure in ferritic steels and the substructure is composed of cell structure [26]. The cell structure size decreases with the increment of applied stress [26]. If the microstrain period is related to this cell structure, it will decrease as the tension process proceeds. However, Figure 9a shows the opposite tendency. This means that this cell structure probably has no significant influence on the microstrain period.

The average peak values of the positive strain localization ($\epsilon_{x,p(+)}$) and the negative strain localization ($\epsilon_{x,n(-)}$) are given in Figure 9b. Both the $\epsilon_{x,p(+)}$ and $\epsilon_{x,n(-)}$ increase with an increase in the applied stress. Normalizing the amplitude of the periodic microstrain by the macrostrain is as follows: the peak value of the positive strain localization ($\epsilon_{x,p(+)}$) divided by the macrostrain ($\epsilon_{x,p(+)/macro}$ and the peak value of the negative strain localization ($\epsilon_{x,n(-)}$) by the macrostrain ($\epsilon_{x,n(-)/macro}$) (Figure 9c). We denote the ratio of the microstrain divided by the macrostrain as the strain concentration factor. If there is no microstrain heterogeneity, the strain concentration factor should be unity. The larger the deviation from unity, the greater the degree of strain concentration. Figure 9c shows that, apparently, at small strains, the absolute value of the strain concentration factor is large; at large strains, the strain concentration factor tends to flatten. For positive stain localization, the maximum tension strain concentration factor decreases from ~6 to ~2 as the tension test proceeds. Negative strain localization exhibits a similar decaying tendency. The compressive strain, even about four times the macrostrain, is present at small strain levels. Ravindran et al. [22] found that compressive strain was present in a low carbon steel subjected to 4.23% plastic strain. The curves of the strain concentration factor versus the macrostrain for positive strain localization and negative strain localization are nearly symmetric with respect to a horizontal line whose strain concentration factor is equal to 1.

It is assumed that each grain has the same deforming ability for a given phase. Because a pure single crystal is correctly oriented and suitably stressed, it deforms along the same crystal orientation, and as a result, deforms in a fairly uniform way [27]. However, in a pure polycrystal, each grain deforms in a different direction because of different crystal orientations, and medium discontinuity is apt to form at the grain boundaries [1]. Of course, grains inevitably accommodate each other to avoid discontinuity by dislocation movement and storage around the grain boundaries. The accommodation between adjacent grains produces strain localization at the grain boundaries. Dislocation also begins to move along a certain plane in grains where there are crystal planes as nearly parallel to the shear stress as possible. Dislocation movement later spreads to grains that are not so favorably oriented and, lastly, to the worst-oriented grains [2]. This dislocation storage in grains contributes to the strain localization in the interior of grains [1]. FEM calculation indicates that nonuniform deformation and strain localization naturally arise in polycrystals as a consequence of crystallographic slip [3].

Figure 9c shows that strain localization is most significant at small macrostrains. This means that the stress concentration is greater at small macrostrains. Figure 5 indicates that this most significant region is in the initial stage of the elastic deformation region. The reason for this phenomenon is unclear. Stress concentration has been studied widely on a macroscopic scale, and many results have been recorded. We try to use these results to understand this phenomenon. It is known that a high stress concentration is present in the region ahead of a notch. When the notch radius tends to zero, the stress ahead of the notch approaches infinity if the material is pure elastic [28]. However, pure elastic is not present in real steel. Therefore, the stress ahead of a notch in a real steel cannot be infinity due to the presence of yielding. This result indicates that plastic deformation can relax the stress concentration. In Figure 9c, as the macrostrain increases, the fraction of plastic deformation increases,
naturally decreasing the degree of the stress concentration. Microstrain concentration within grains probably provides the source of macroscopic yielding or the initiation site of fracture.

**Figure 9.** Dependence of the microstrain period, microstrain amplitude and strain concentration factor on the macrostrain. (a) average period versus the macrostrain; (b) average peak values of the positive and negative microstrain localizations versus the macrostrain; (c) average strain concentration factors of the positive and negative microstrain localizations versus the macrostrain. Error bar is the standard error. The vertical dotted red line corresponds to the noise-to-strain ratio of 0.5.
4. Conclusions

The tensile deformation of 0.05% C polycrystalline steel within the macroscopic elastic region was observed in situ, and the microstrain within grains was measured using two-dimensional DIC. The conclusions obtained are as follows:

(1) Microstrain distribution within grains in the elastic deformation region was pseudo-periodic. The period increased with the increase of applied stress, tending to approach the grain size;
(2) The degree of microstrain concentration decreased as the tensile test proceeds. The compression microstrain, even approximately four times that of the macrostrain, was present at small strain levels;
(3) Great misorientation angle and significant difference in the elastic modulus between adjacent grains promoted microstrain concentration at grain boundaries. However, microstrain concentration did not inevitably occur even if both the misorientation angle and the difference in the elastic modulus were great. Strong concentration of microstrain provided easy sites for microyielding.

Author Contributions: Each author contributed equally to this work. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Conflicts of Interest: The authors declare no conflict of interest.

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