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Abstract

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Full Text

Preamble

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Plastic Strain Heterogeneity and Work Hardening of Ni Single Crystals

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Abstract

The work hardening behavior of Ni single crystals was investigated from the perspective of strain heterogeneity by acquiring full-field strain information through digital image correlation. First, a digital image correlation method suitable for characterizing single crystal deformation was proposed to accurately obtain strain fields. Tensile test results demonstrate that the plastic strain in Ni single crystals exhibits significant localization characteristics, which are closely related to the formation and development of slip bands. Based on the evolution characteristics of the strain field, three deformation regimes can be identified, and they show a one-to-one correspondence with the three work hardening stages of the material. Within the framework of dislocation theory, the intrinsic connection between them has been reasonably explained and well verified through experimental observations of the material's microstructural evolution.

KEY WORDS Ni single crystal, work hardening, plastic deformation, digital image correlation, heterogeneity

Introduction

The phenomenon of work hardening in materials during plastic deformation has long attracted the attention of metallurgists. However, it was not until the 1930s, with the introduction of dislocation theory [?], that the microscopic mechanism of work hardening was revealed. According to classical dislocation theory, plastic deformation in materials at low temperatures is caused by the long-range motion of dislocations under applied stress. This process is typically accompanied by dislocation multiplication, interactions between dislocations, and an increase in dislocation density. Building upon this theoretical foundation, Schmid and Boas [?] proposed the famous critical resolved shear stress law, which states that slip motion occurs along specific slip planes and directions when the resolved shear stress on a given slip system reaches a critical value. With the establishment of these fundamental theories of crystal plasticity, people have gained a deeper understanding of material work hardening behavior and conducted extensive experimental research. Notably, the three stages of work hardening have been universally confirmed in fcc crystals [?], attracting widespread interest among scholars.

Dislocation observations require microscopic-scale techniques, which is why transmission electron microscopy (TEM), scanning electron microscopy (SEM),

and atomic force microscopy (AFM) have been widely applied in dislocation research. Through these microscopic observation methods, dislocation structures and their evolution during plastic deformation have been described. Research shows that after undergoing plastic deformation, dislocation distribution is often neither uniform nor random, but rather exhibits very localized characteristics (especially under multiple slip conditions) [?]. This phenomenon is not difficult to explain, because dislocation motion, generation, and annihilation are ultimately governed by strain, which is not uniformly distributed at either macroscopic or microscopic scales [?, ?, ?, ?, ?]. Although the basic characteristics of crystal plasticity have long been well known, few scholars have conducted in-depth and systematic studies on the relationship among plastic strain heterogeneity, microstructural evolution, and macroscopic mechanical behavior.

As early as the 1970s, Takamura [?] pointed out that scholars' excessive focus on dislocation structure itself had led to the neglect of strain heterogeneity—a fundamental characteristic of plastic deformation. His subsequent research [?] demonstrated that plastic strain heterogeneity directly affects the orientation of dislocation motion and promotes the formation of deformation bands, thereby causing material work hardening. Due to limitations in experimental technology at that time, Takamura [?] could only qualitatively describe this phenomenon through surface morphology observations of deformed specimens (such as deformation band formation) rather than quantitatively characterizing plastic deformation heterogeneity through strain fields.

Today, the emerging digital image correlation (DIC) method [?, ?, ?, ?] enables real-time measurement of displacement and strain fields. This method offers advantages such as full-field and non-contact measurement and has been widely applied in materials mechanics research. Experimental studies [?] have shown that the DIC method provides excellent strain measurement accuracy, and real-time strain field acquisition holds high practical value for describing material mechanical behavior. As the technology continues to mature, DIC has been increasingly applied to meso- and micro-scale research [?, ?, ?, ?, ?, ?, ?], often combined with classical microscopic observation methods [?, ?] and crystal plasticity finite element simulations [?, ?], demonstrating its important value in multi-scale experimental studies. Regarding the work hardening problem of interest in this study, the potential connection among strain heterogeneity, macroscopic mechanical behavior, and microstructural evolution could serve as a breakthrough for deeply understanding material work hardening mechanisms. The DIC method provides the necessary technical means for this research, making it possible to re-examine work hardening from the perspective of strain heterogeneity through experimental studies.

To achieve these research objectives, this work employs the DIC method as the primary research tool to conduct experimental studies on the plastic mechanical behavior of [001]-oriented Ni single crystals. The focus is placed on crystal plasticity research based on strain fields—specifically, the correlation between

strain field evolution and slip system activation, as well as the intrinsic connection among strain heterogeneity, work hardening stages, and microstructural evolution.

1.1 Materials and Experiments

The research material was a 99.999% pure Ni single crystal. The single crystal was obtained by the Bridgman crystal growth method and subsequently machined into a standard tensile specimen with geometric dimensions shown in [Figure 1: see original paper].

The specimen surface was oriented at (100), and the length direction (i.e., the tensile axis) was along [001]. Since Ni has an fcc crystal structure, there are theoretically 12 slip systems of the $\{111\}\langle 110\rangle$ type. Based on the crystal orientation of the current specimen, purely from a crystallographic geometry perspective: eight slip systems would experience the same magnitude of loading during tensile testing, with a Schmid factor of 0.4, making them potentially active slip systems. The remaining four slip systems have a Schmid factor of 0 and therefore would not be activated during tensile testing. Among the eight potentially active slip systems, four would intersect the (100) surface, resulting in two sets of slip traces (or slip bands) on the specimen surface—essentially the intersection of slip planes from two slip system combinations with the specimen surface. These crystallographic predictions would be verified in subsequent mechanical experiments and observations.

Before tensile testing, the specimen surface needed to be coated with a black-and-white speckle pattern, which is a special requirement of the DIC method for the test object surface. During mechanical testing, these speckles serve as carriers of material deformation information, deforming together with the material, and their grayscale distribution characteristics facilitate digital image recognition, registration, and correlation processing.

Uniaxial tensile experiments were conducted at room temperature using a Kammrath & Weiss Tensile/Compression Module 5kN micro-tensile testing machine. The tensile tests were displacement-controlled at a speed of 19 m/s, corresponding to an axial strain rate of $2 \times 10^{-3} \text{ s}^{-1}$. When the total strain reached 0.22, the loading process was stopped and unloading began. Simultaneously with the tensile experiment, a DIC system was used for real-time observation of specimen surface deformation. A DIC system generally consists of a CCD/CMOS industrial camera, optical lens, white light source, and DIC analysis software. The camera used in this experiment was an Elphel NC353L CMOS camera with $2592 \times 1936 \text{ pixels}$. *When equipped with a Tamron zoom lens, the spatial resolution reached $10.8 \mu\text{m} \times 10.8 \mu\text{m}$ per pixel.* The camera sampling frequency was set to 15 frames/s, which effectively guaranteed the “real-time” recording of specimen surface deformation given the relatively slow tensile test speed.

Before introducing the DIC method, it is necessary to first observe the surface

morphology of the deformed single crystal specimen, as this directly affects the data processing strategy selection in the subsequent DIC method. The surface morphology after tensile testing is shown in [Figure 2: see original paper]. Two sets of slip bands were observed on the specimen surface, consistent with the crystallographic predictions. However, one set of slip traces was more pronounced than the other (wider slip bands, higher steps), indicating that the activated slip systems were not uniformly loaded under actual conditions. For convenience, the more pronounced set is referred to as primary slip bands, and the other as secondary slip bands. [Figure 2: see original paper] reveals that: (1) the specimen surface exhibits significant non-uniform, discontinuous deformation characteristics; and (2) primary slip bands dominate this non-uniform deformation. Both points are closely related to the design of the DIC method.

1.2 DIC Method

The DIC method is essentially a novel optical measurement technique based on modern digital image processing and analysis, which obtains surface deformation (displacement and strain) information by analyzing digital images of an object surface before and after deformation.

During tensile experiments, the CMOS camera captured digital images of the specimen surface during deformation. Each digital image corresponds to a scalar function of spatial coordinates, providing grayscale values for each discrete pixel point x . The image before loading is typically called the “reference image,” denoted by f ; subsequent images are called “deformed images,” denoted by g . Under the optical flow assumption [?], these satisfy the following equation:

$$f(x) = g(x + u(x))$$

where a displacement field u exists between f and g . The transfer function, also called the shape function, is used to represent the transformation from f to g . The goal of DIC is to find this transfer function $\Phi(x)$ between two known images f and g . This is typically an ill-posed problem because relying solely on grayscale level information is insufficient to uniquely determine the displacement vector at each pixel point. Therefore, the problem requires regularization by constraining the form of the transfer function, usually parameterized as $\Phi(x)$, containing N scalar parameters α . Thus, the DIC problem can be transformed into an optimization problem [?]:

$$\hat{\alpha} = \arg \min C(\alpha)$$

where the correlation function C measures the similarity between the reference image f and the transformed image $g(\Phi(x))$. Different forms of correlation functions exist; this work adopts the definition based on the cross-correlation coefficient between two functions [?]:

$$C = 1 - \frac{\int f \cdot g \, d\Omega}{\left(\int f^2 \, d\Omega\right)^{1/2} \cdot \left(\int g^2 \, d\Omega\right)^{1/2}}$$

where R represents the image correlation analysis region, and \bar{f} and \bar{g} denote

the average values within region R.

By solving equation (3) through optimization algorithms and determining $\Phi(x)$, the displacement at each pixel point can be obtained from equation (2), yielding the displacement field. Under the classical “continuum assumption,” the strain field is typically obtained through finite difference of displacements [?]. However, this method is not suitable for single crystal materials, as [Figure 2: see original paper] has revealed that plastic deformation exhibits significant discontinuity features due to slip band formation. Studies [?, ?] have shown that dislocation slip bands often display much higher strain levels than surrounding regions. Therefore, this work proposes a data processing strategy suitable for describing single crystal plastic deformation to ensure the accuracy of displacement and strain evaluation by the DIC method under discontinuous medium conditions.

1.3 Displacement and Strain Calculation Under Discontinuous Medium Conditions

In DIC calculations, the transfer function $\Phi(x)$ covering region R can be decomposed into multiple independent, local transfer functions (or shape functions), i.e., $\Phi(x)$ can be parameterized within a small region D_0 centered at x_0 [?]:

$$\Phi(x) = x_0 + \sum \alpha_0 e + \sum \alpha_1 (x - x_0)_i e_i$$

where e_i represents the unit vector along direction i in the image, and x is the coordinate of pixel point x , with $i \in \{1,2\}$. Equation (5) typically adopts a first-order form, including translation α_0 and first-order derivatives of deformation α_1 , totaling 6 parameters to determine a local shape function. This data processing strategy is generally called the “local approach,” as opposed to the “global approach” [?], which does not use partitioning but determines a unique shape function covering the entire region R. Obviously, the latter is not suitable for describing mechanical behavior with significant discontinuous deformation features.

When using the local approach for DIC calculations, the image to be processed must first be divided into several small local regions D , generally called correlation windows or image subsets. The selection of correlation windows is a crucial step in DIC, directly affecting the accuracy of displacement and strain field evaluation and their mechanical interpretation. Generally, the larger D is, the more overall (or macroscopic) the described deformation; the smaller D is, the more local the described deformation.

D can be defined as a square region of H pixel \times H pixel. To better capture slip behavior on the single crystal surface, the size of D should not exceed the average slip band width \bar{d} . Observation of the deformed Ni single crystal specimen surface revealed that the average slip band width was approximately 200 nm, corresponding to about 19 pixels in the digital image:

$$\bar{d} = 19 \text{ pixels}$$

Through testing, when $H = 16$ pixels (i.e., D size of $16 \text{ pixel} \times 16 \text{ pixel}$), the obtained displacement field achieved satisfactory results: it could capture slip-related local displacement while avoiding low signal-to-noise ratios caused by excessively small D . After determining D size, optimization algorithms can determine the parameters of each local shape function, thereby calculating the displacement field for the entire image. Applying this method to each captured image yields the displacement field on the specimen surface at different loading moments. [Figure 3: see original paper]a and b show the axial displacement field (u_y) and transverse displacement field (u_x) corresponding to the final deformation state, respectively. The results show that displacement variation in space is not continuous but exhibits band-like discontinuity along the 45° direction (the crystal's primary slip direction), indicating close correlation with slip system activation. Notably, only one set of slip bands (the primary slip bands) appears in both axial and transverse displacement fields, with no secondary slip bands present. This aligns with surface morphology observations, where primary slip bands dominate the deformation, while secondary slip bands cannot be reflected in the overall displacement field due to their relatively small local displacement magnitude.

The discontinuity in the displacement field essentially results from dislocation slip motion on the crystal surface, which macroscopically manifests as slip band formation. Slip bands introduce a displacement jump across the slip plane, creating a discontinuity, while the continuity of deformation within slip bands (or non-slip bands) remains unaffected. To ensure strain calculation is not influenced by this medium discontinuity, slip bands (and slip band intervals) can be treated as basic analysis units, with each unit assumed to be an independent continuous medium where displacement is continuous within each unit. Under this assumption, data within each analysis unit can be processed independently. This work uses quadratic polynomials to fit displacement data within each unit, obtaining functions F_{u_y} and F_{u_x} for axial and transverse displacements, respectively:

$$\begin{aligned} F_{u_y} &= a_0 + a_1x + a_2y + a_3x^2 + a_4y^2 + a_5xy \\ F_{u_x} &= b_0 + b_1x + b_2y + b_3x^2 + b_4y^2 + b_5xy \end{aligned}$$

where a_k and b_k ($k = 0, 1, \dots, 5$) are two sets of constants.

Within each analysis unit, three plane strain components can be obtained by differentiating these functions [?]: axial strain ϵ_{yy} , transverse strain ϵ_{xx} , and shear strain ϵ_{xy} :

$$\begin{aligned} \epsilon_{yy} &= F_{u_y} / y = a_2 + 2a_4y + a_5x \\ \epsilon_{xx} &= F_{u_x} / x = b_1 + 2b_3x + b_5y \\ \epsilon_{xy} &= \frac{1}{2}(F_{u_y} / x + F_{u_x} / y) = \frac{1}{2}(a_1 + a_5y + 2a_3x + b_2 + b_5x + 2b_4y) \end{aligned}$$

In this study, the von Mises equivalent strain ϵ_{eq} [?] is used to characterize plastic deformation on each slip plane of the single crystal. Here, ϵ_{eq} can be calculated from the three strain components:

$$\epsilon_{eq} = \sqrt{(\epsilon_{xx}^2 + \epsilon_{yy}^2 + 2\epsilon_{xy}^2)}$$

By processing each analysis unit sequentially, the corresponding von Mises equivalent strain field can be obtained. Applying the same processing to all images captured during the tensile test yields the evolution of the von Mises equivalent strain field on the specimen surface, enabling in-depth analysis of the plastic deformation behavior of Ni single crystals.

2 Experimental Results and Analysis

[Figure 4: see original paper] shows the axial stress-strain curve of the specimen during tensile testing, along with the von Mises equivalent strain fields at moments A~F. The strain field evolution reveals that at the initial deformation stage, no slip-related strain localization features were observed, as shown in strain field A. However, as loading gradually increased, strain became extremely non-uniform and concentrated in specific banded regions—namely, the activated slip bands—as shown in strain fields B and C. In the final deformation stage, the heterogeneous morphology of the strain field tended toward stability, as shown in strain fields D~F.

Based on these strain field evolution characteristics, the deformation process of the Ni single crystal specimen can be categorized into three deformation regimes, with representative von Mises equivalent strain fields shown in [Figure 5: see original paper]. Their main features can be summarized as follows:

- (1) **Deformation Regime I—Uniform Deformation Mechanism**
Strain remains at a very low level overall, with no particular strain heterogeneity directly related to slip behavior, as shown in [Figure 5: see original paper]a. However, this does not mean that slip systems are not activated or that strain distribution is uniform at a more microscopic scale; it only indicates that at the current scale, strain exhibits a relatively uniform distribution characteristic.
- (2) **Deformation Regime II—Localized Deformation Mechanism**
The transition from regime I to regime II appears rather abrupt: the emergence of a slip band rapidly changes strain field uniformity, while strain in other regions remains relatively uniformly distributed, as shown in [Figure 5: see original paper]b. When the first slip band appears, the average ϵ_{eq} within the slip band reaches 2.9%, while the mean ϵ_{eq} across the entire strain field is only 1.1%. Following the formation of the first slip band, new slip bands continue to emerge, showing significantly higher strain than surrounding regions. This dynamic process continues until the last slip band forms and the crystal surface morphology stabilizes.
- (3) **Deformation Regime III—Steady-State Deformation Mechanism**
The steady-state deformation mechanism corresponds to the final stage of crystal plastic deformation, where the strain field distribution pattern

tends toward stability. As shown in [Figure 5: see original paper]c, slip bands (light-colored) and non-slip bands (dark-colored) can be clearly distinguished in most regions, as the former typically shows significantly higher strain than the latter.

To describe these three deformation regimes more intuitively and simply, slip bands (and slip band intervals) are again treated as basic analysis units, with the average strain within each unit used to characterize the strain level of that unit. Thus, the two-dimensional strain fields shown in [Figure 5: see original paper] can be described in one-dimensional curve form, i.e., the distribution of ϵ_{ij} across different units under the three deformation regimes, as shown in [Figure 6: see original paper]. In this example, there are 25 units in the analysis region, with unit numbers denoted by i . The strain distribution curves in [Figure 6: see original paper] show that the process from uniform to localized to steady-state strain distribution under the three different deformation regimes is well captured, indicating that this description method captures the main features of the deformation mechanisms.

Based on this analysis method, this work proposes a sensitive index s_B to quantitatively determine the three deformation regimes. s_B characterizes the standard variance of ϵ_{ij} distribution across N units and is defined at time t as:

$$s_B(t) = \sqrt{\left[\frac{1}{N} \sum_{i=1}^N (\epsilon_{ij}(t) - \bar{\epsilon}(t))^2 \right]}$$

$$\bar{\epsilon}(t) = \frac{1}{N} \sum_{i=1}^N \epsilon_{ij}(t)$$

[Figure 7: see original paper] shows the evolution of s_B during the tensile test and the determination of the three deformation regimes based on s_B trends. In regime I, s_B is small and remains nearly constant; in regime II, s_B shows a rapid growth trend; in the final regime III, s_B remains at a high level without obvious monotonic increase or decrease. Consequently, the shear strain γ ranges corresponding to regimes I, II, and III are determined as: 0~5.0%, 5.0%~23.4%, and 23.4%~55.0%. Note that the shear strain here is converted from axial strain using the Schmid factor, and shear strain is used for description to facilitate comparison between the deformation regimes proposed in this work and the classically defined work hardening stages of single crystals.

Based on the shear stress-strain (τ - γ) curve of the specimen during tensile testing, the work hardening stages during plastic deformation can be determined by calculating the work hardening rate ($= d\tau/d\gamma$) as a function of γ [?]. The τ - γ and $d\tau/d\gamma$ curves for the Ni single crystal specimen in this experiment, along with the three work hardening stages determined from the latter's trend, are shown in [Figure 8: see original paper]. The three work hardening stages of the Ni single crystal are: 0~6.4%, 6.4%~24.0%, and 24.0%~55.0%. This result indicates that the three work hardening stages of the material correspond one-to-one with the three deformation regimes proposed above. In terms of corresponding strain ranges, the boundary between deformation regimes I and II is $\gamma = 5.0\%$, which differs by only 1.4% from the boundary between hardening stages I and II at

$\gamma = 6.4\%$; the boundary between deformation regimes II and III at $\gamma = 23.4\%$ differs by only 0.6% from the boundary between hardening stages II and III at $\gamma = 24.0\%$. Considering potential errors and uncertainties in the analysis methods themselves, especially for determining work hardening stages, these data are considered sufficient to reveal a one-to-one correspondence between deformation regimes and work hardening stages.

Regarding the one-to-one correspondence between deformation mechanisms and work hardening stages for pure Ni single crystals under tensile loading, this work attempts to provide a reasonable explanation for their intrinsic connection within the framework of dislocation theory.

First, for single crystal materials, the classically defined work hardening stage I, the “easy glide” stage, only applies to single slip conditions and does not appear under multiple slip conditions. For convenience, the term “stage I” is still used here to represent the stage before “linear hardening” appears. The corresponding uniform deformation regime I can be understood as the stage before macroscopic slip behavior occurs. In this regime, without the formation of macroscopic slip bands, the microscopic plastic deformation caused by slip system activation has not yet brought the material into a significant work hardening stage, and the corresponding strain field exhibits relatively uniform distribution characteristics.

Work hardening stage II, also known as the linear hardening stage, is generally understood as resulting from multiple slip generating numerous dislocations, with dislocation interactions significantly increasing the resistance to dislocation motion, thus producing pronounced work hardening effects [?, ?]. The corresponding deformation regime II shows that the strain field exhibits significant localization characteristics during this stage, with strain concentration regions corresponding to slip bands. This indicates that at the microscopic level, dislocation distribution is also extremely non-uniform, and strain localization is precisely the kinematic manifestation of dislocation motion on the crystal surface at the macroscopic level. Furthermore, [Figure 7: see original paper] shows that the index s_B measuring strain heterogeneity exhibits an approximately linear growth trend in regime II, which matches the “linear” hardening characteristics shown in the τ - γ and $-\dot{\gamma}$ curves in [Figure 8: see original paper].

The final work hardening stage III is the parabolic hardening stage, typically explained as the reduction in work hardening rate caused by cross-slip effects [?], with dislocation density entering a saturated state. The corresponding deformation mechanism is steady-state deformation regime III. In this regime, the strain distribution pattern tends toward stability, with no new strain concentration bands appearing, all indicating that no large numbers of new dislocation sources are generated in this stage. The saturation of dislocation density also matches the stable level of s_B in this regime.

To verify the above theoretical analysis, this work observed the microstructure of pure Ni single crystal specimens at different deformation levels ($\gamma = 4\%$,

11%, 30%, 50%, 70%, 83%) using JEM-2010 TEM. Experimental results show that under multiple slip conditions, different types of dislocation configurations form with increasing load levels, such as dislocation pile-ups, Lomer-Cottrell locks, dislocation cells, and dislocation walls. Due to the highly non-uniform distribution of dislocations, for convenience in statistically measuring dislocation density, they are simplified into two categories: Type 1 is the high dislocation density corresponding to relatively hard dislocation walls, denoted as ρ_w ; Type 2 is the low dislocation density corresponding to relatively soft dislocation cells, denoted as ρ_c . The average dislocation density ρ_t of the material can be obtained by combining these two densities:

$$\rho_t = f_w \rho_w + (1 - f_w) \rho_c$$

where f_w represents the volume fraction of dislocation walls, and $(1 - f_w)$ represents the volume fraction of dislocation cells.

Based on TEM observations of dislocation structures, the parameters ρ_w , ρ_c , and f_w can be estimated using a classical statistical method [?], and the material's average dislocation density ρ_t can then be obtained from equation (15). [Figure 9: see original paper] shows the variation trends of these three dislocation densities with strain level. The results indicate that all three dislocation densities exhibit very similar variation patterns: rapid growth during work hardening stage II and a relatively saturated state during work hardening stage III. This result is consistent with the predictions based on dislocation theory and further confirms that the intrinsic connection between the deformation regimes proposed in this work and the traditional work hardening stages can be reasonably explained through dislocation theory.

Conclusions

- (1) The evolution of strain fields is closely related to the activation of crystal slip systems and the formation and development of slip bands.
- (2) Based on quantitative analysis of von Mises equivalent strain field evolution characteristics, three distinct deformation regimes can be identified: uniform deformation regime, localized deformation regime, and steady-state deformation regime.
- (3) These three deformation regimes correspond one-to-one with the three work hardening stages of single crystals under classical definitions (easy glide stage, linear hardening stage, and parabolic hardening stage). The intrinsic connection between them has been reasonably explained through dislocation theory and verified by dislocation density measurements at the microstructural scale.

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