Tensile Behavior of Fine-grained Steels

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With decreasing of grain size in ferritic steels, Lüders elongation becomes larger while work-hardening is lowered, finally resulting in loss of uniform elongation. This drawback can be overcome by introducing the second phase like martensite or metastable austenite. The improvement of strength and uniform elongation balance by the second phase can well be estimated by applying the secant method of micromechanics approach. The stress partitioning between two constituents brings high work-hardening, which is verified by in situ neutron diffraction. The influences of strain rate and temperature are described by using the Kocks–Mecking model. It is found that the grain refinement and the above stress partitioning contribute mainly to the athermal stress component of flow stress. Hence the tensile properties obtained at a high speed deformation like 10³/s is excellent in fine-grained multi-phase steels. As an example of ultrafine-microstructure with 20–30 nm in size, the tensile behavior of severely drawn pearlite steel wires with tensile strength larger than 4 GPa is investigated. In spite of such ultra-high strength, the wire deforms plastically by dislocation motion resulting in dimple fracture. The strengthening consists of isotropic hardening due to microstructure refinement and anisotropic hardening caused by residual intergranular stresses which are determined by neutron diffraction.

KEY WORDS: ultrafine grained steel; Lüders deformation; necking; work-hardening; TRIP.

1. Introduction

Effect of grain size on the yield strength as well as flow stress in steels are generally described by the Hall–Petch equation1,2)

\[ \sigma_s = \sigma_0 + kD^{1/2} \]

where \( \sigma_s \) and \( D \) refer to the yield strength or flow stress and average grain size, respectively, and \( \sigma_0 \) and \( k \) are constants. The above empirical equation was originally established for the experimental data in the grain size region larger than 10 \( \mu m \).3) According to a textbook written by Leslie,4) the grain size range was extended to a submicron range based on Miller’s work in 1972.5) It has been recognized that the minimum size achieved by conventional thermo-mechanical control process (TMCP) is 5 \( \mu m \) for low alloyed steels. Recently sub-micron grained steels have been made by advanced TMCP encouraged by national projects.6,7) In this review, tensile behavior of steels with fine grain sizes are discussed focusing on (1) shape of stress–strain curves and Lüders strain, (2) improvement of ductility by using the second phase, (3) effect of grain size on strain rate dependence of flow stress and (4) deformation mechanism of ultrafine grained steels.

2. Shape of Stress–Strain Curves and Lüders Strain

With decreasing of grain size, various types of stress–strain curves are observed. Referring to Miller,5) Tsuji et al.8) and Yu et al.,9) stress–strain curves are classified into four types as illustrated in Fig. 1. The definition of “yield strength” is not consistent in different types and therefore the constant \( k \) in Eq. (1) differs from type to type. This is because the definition of the yield strength is different depending on the type.10) Hence, the difference in the curve type should be taken into consideration for discussion how grain size affects stress–strain behavior. Work-hardening is also an important subject because the ultrafine-grained alloys have a drawback that the uniform elongation decreases with a decrease in grain size.

Tsuchida et al.10) and Narui et al.11) have studied tensile behavior of low carbon ferrite–cementite (FC) steels with

![Fig. 1. Schematic illustration for classification of nominal stress–strain curves.](https://example.com/fig1.png)
grain size ranging from 0.47 to 13.6 μm. The specimens were prepared through groove rolling for JIS-SM490 steel (Fe–0.15C–0.3Si–1.4%Mn–0.009P–0.001S in mass%). The microstructures of the FC steels for ferrite grain size of 1.5 μm and 0.47 μm are shown in Fig. 2 where white area is corresponding to cementite. As was reported by Yin et al., high angle grain boundary occupies approximately 70% in the 0.47 μm specimen. Pole figures determined by X-ray diffraction for the 0.47 μm specimen showed weak 110 fiber texture. Since necking occurs during the Lüders deformation, work-hardening is not observed. Hence, compression test was performed for a cylinder specimen with 6 mm diameter and 10 mm length and the results were shown in Fig. 3(b). In the compression test, Lüders deformation was hardly observed probably due to the short gauge length of the specimen. Here the main concern includes the deformation behavior in tension after the yielding and the relationship between Lüders deformation and the onset of necking in the 0.47 μm specimen. True stress–strain (σ–ε) relationship was determined from the data obtained by compression test and then work-hardening rate (dσ/dε) was calculated (see Fig. 3(c)). Here, dσ/dε–ε curve (dashed line) and σ–ε curve (solid line) crosses at a very small strain, i.e. the conventional criterion for the onset of necking. Then, the aspect of Lüders deformation behavior was observed with an optical microscope for the tensile specimens and results are shown in Fig. 4. As seen, the 1.5 μm specimen in (a) looks similar to usual cases. Compared to this, the 0.47 μm specimen in (b) shows sharp multi-bands. The specimen in Fig. 4(b) was examined with a confocal laser scanning microscope to measure the height change on their surface. As seen in Fig. 5, three regions are recognized, i.e., non-deformed region, Lüders band region and necked region. The change in specimen thickness within the band was about 60 μm (30 μm on one side). Taking the initial thickness of the specimen of 1.0 mm into account and neglecting the change in specimen width, the 60 μm reduction is corresponding to tensile strain of approximately 13.2% which must be regarded as Lüders strain. The work hardening becomes extremely small leading to the loss of uniform elongation in ultrafine grained steels as well as aluminum with grain size of less than approximately 1.0 μm, where the characteristic shape of stress–strain curve appears. In such ultrafine grained steels, necking occurs during Lüders deformation after the yield drop.

Tsuchida et al. have found the Lüders strain is summarized by one parameter, i.e., the work-hardening rate just after the end of Lüders deformation as is presented in Fig. 6. In case of type II stress–strain curve, the Lüders strain is speculated to be very large. On the other hand, the plastic instability condition to bring necking in tension is described by Eq. (2).

\[
\sigma > \frac{d\sigma}{d\varepsilon} \ \ \ \ \ \ \ \ \ (2)
\]
where $\sigma$ and $\varepsilon$ refer to true stress and true strain, respectively. Hence, necking must possibly take place during the Lüders deformation in materials showing type II stress–strain curve. The Lüders strain and work-hardening rate for the 0.47 mm specimen are 3.2% and 1 000 MPa, respectively, showing a good agreement with the trend in Fig. 6. Only one parameter of the work-hardening rate ($d\sigma/d\varepsilon$) determines the magnitude of Lüders elongation. A very interesting point is a good correspondence between Lüders strain and $d\sigma/d\varepsilon$, which are hardly influenced by other influential factors including test temperature, strain rate, grain size etc.\(^1\) In Fig. 6, the following relationship is found to hold approximately.\(^1\)  

$$\varepsilon_L \times (d\sigma/d\varepsilon)_{LYS} = \Delta$$  

where $\varepsilon_L$ refers to the Lüders strain, $(d\sigma/d\varepsilon)_{LYS}$ the work-hardening rate at the lower yield stress (LYS) and $\Delta$ a constant. There exist three areas during the propagation of the band: (a) already plastically deformed area, (b) Lüders band front, and (c) virgin area without any plastic deformation. The deformed area is work-hardened to the stress level at LYS corresponding to the Lüders strain. Therefore, Eq. (3) is postulated to indicate geometrical stress balance in a non-uniformly deformed specimen, where work hardening is a controlling factor.

3. Improvement of Ductility by Using the Second Phase

A method to increase work hardening is needed in order to prevent the loss of uniform elongation in an ultrafine grained steel. One of the potential ways is introduction of the hard second phase. Tsuchida et al. have calculated strength and elongation balance by using the secant method and claimed that introduction of the hard second phase particles is effective to increase not only tensile strength but also uniform elongation in case of fine grained steels.\(^1\) Concerning the details on the secant method, see Refs. 16)–20). An example of calculations for ferrite–pearlite steels is shown in Fig. 7.\(^1\) When the ferrite grain size decreases, the tensile strength would increase while uniform elongation decrease. If the harder phase is introduced, the strength and elongation balance would be improved as seen in Fig. 7. To be noted here is that both strength and elongation increase in a fine grain region. The secant method was also applied to deformation of TRIP steels; metastable austenitic steel, Ni bearing steel for cryogenic use and TRIP assisted multi-structure steel.\(^2\) For industrial production, TRIP assisted fine grained dual phase steel must be promising.

Stress partitioning behavior in two-ductile-phase steels during tension and/or compression deformation can well be monitored by using neutron diffraction.\(^2\) In a TRIP assisted multi-structure steel for automobile use, carbon enriched austenite is found to be harder than ferrite.\(^2\) The austenite, therefore, plays the hard phase and moreover transforms to martensite during tensile deformation, which is very effective to increase work hardening. Hence, ultrafine grained ferrite–austenite steels are now the target to be developed. There have already been a few TMCP procedures to be able to obtain such a microstructure. One of the examples\(^11\) is described below.

Ultrafine grained ferrite–austenite (FA) steel was prepared by tempering after cold rolling of martensite plates quenched from 1 273 K for an Fe–17.2Ni–0.2C alloy.\(^1\) After austenitization at 1 273 K for 3.6 ks followed by water quenching, specimens were subjected to rolling with an accumulated area reduction of 70% at room temperature and
tempered at 823 K for 18 ks. Tensile specimens with 30 mm in gauge length, 3 mm width and 1 mm thickness were made along the rolling direction and then polished using colloidal silica. Static tensile test was carried out at room temperature with a strain rate of $2.8 \times 10^{-3} \text{s}^{-1}$. The microstructure of FA steel is presented in Fig. 8, where the austenite volume fraction is approximately 46% determined by X-ray diffraction. EBSD results showed that the grain size was about 0.52 $\mu$m. Nominal stress–strain curve is shown in Fig. 9. The FA steel shows tensile strength of 1.1 GPa and uniform elongation of 27%. According to the classification of Fig. 1, the FA steel showed type III in spite of ultrafine grains. Similar results have been obtained in other ultrafine grained dual phase TRIP steels. The pioneering report was firstly made by Miller. Hence, the introduction of metastable fine austenite grains is one method to overcome the drawback of ultrafine grained ferrite steels.

In order to examine what is happening during the Lüders deformation, in situ neutron diffraction was carried out for the FA steel. The measuring method of neutron diffraction is the same with the previous work. The angular dispersion neutron diffraction with a wave length of 0.1786 nm was performed during stepwise tensile testing using a diffractometer for residual stress analysis (RESA) at the Japan Atomic Energy Agency (JAEA). The gauge volume of neutron diffraction was $5 \times 3 \times 1 \text{mm}^3$ (slit width: 5 mm) so that the averaged lattice strains in a bulk specimen was evaluated. Neutron diffraction profiles were obtained for (110) ferrite, (200) ferrite, (111) austenite, and (200) austenite. Peak shift $\Delta \theta_{hk} (= \Delta \theta_{hk} - \theta_{hk})$ with tensile straining was converted to elastic strain ($\varepsilon_{hk}$) by

$$\varepsilon_{hk} = (d_{hk} - d_{hk}^0) / d_{hk}^0 = - \cot(\theta_{hk}) \Delta \theta_{hk}$$

where $d_{hk}$ and $d_{hk}^0$ refer to (hkl) spacing, and the reference spacing (stress-free), respectively. Figure 10 shows diffraction profiles of austenite (200), the intensity of which was obviously decreased immediately after the Lüders band had propagated at the lower yielding stress (LYS). The (hkl) lattice strain is shown in Fig. 11 as a function of the applied stress. The lattice strain increases linearly with loading in an elastic regime. Then, the ferrite (200) strain increases rapidly, while that in austenite decreases a little with increasing of the applied stress after LYS. It implies that austenite is softer than ferrite. This is different from the case of TRIP assisted multi-phase steel studied before, presumably because the ultrafine grained ferrite matrix is stronger and carbon concentration of austenite is not high, approximately 0.3 mass% in the present steel. The stress partitioning behavior is, therefore, similar to duplex stainless steels. The high work hardening after the Lüders deformation is postulated to be caused by stress-induced martensite.

4. Effects of Strain Rate and Ferrite Grain Size on Flow Stress

Thermal activation mechanism of dislocation motion and dislocation structure evolution are well described by
Kocks–Mecking model.\textsuperscript{24,25} Here, the outline of the model is given to discuss the effect of grain size on strain rate sensitivity of flow stress. Concerning the details of the model and applications to steels, see Refs. 24–28). Flow stress ($\sigma$) is described as a function of temperature ($T$), strain rate ($\dot{\varepsilon}$) and true strain ($\varepsilon$) as follows,

$$\frac{\sigma}{\mu} = \frac{\hat{\sigma}_s}{\mu} + \frac{s_i(T, \dot{\varepsilon})}{\mu_0} + \frac{s_D(T, \dot{\varepsilon})}{\mu_0} \hat{\sigma}_D \quad \text{(5)}$$

where $\mu$ is temperature dependent shear modulus and $\mu_0$ the shear modulus at 0 K. The first term of the right-hand side, $\hat{\sigma}_s$, is the athermal stress which means the yield stress at a temperature above the critical temperature $T_c$. Although $\hat{\sigma}_s$ is sometimes regarded as a material constant, it is expressed as a function of strain in steels; work hardening contains the athermal stress component. The second term and third terms mean two different kinds of obstacles for dislocation motion accompanying relevant thermal activation mechanisms; the second term refers to yielding, i.e., Peierls potential barrier, solid solution hardening, etc., where the mechanical threshold stress, $\hat{\sigma}_s$, is lowered with thermal activation by factor $s_i(\dot{\varepsilon}, T)$; in the third term, $\hat{\sigma}_D$ is a threshold stress to overcome a barrier caused by dislocation–dislocation interactions, which is also decreased by $s_D(\dot{\varepsilon}, T)$. To be noted is that $\hat{\sigma}_D$ increases during deformation by increasing of dislocation density that depends on temperature and strain rate because dynamic recovery takes place. The following equations are frequently used for $s_i(\dot{\varepsilon}, T)$ and $s_D(\dot{\varepsilon}, T)$, respectively.

$$s_i(T, \dot{\varepsilon}) = \left[ 1 - \frac{kT}{g_0 \bar{\varepsilon}_D \mu b^3} \ln \left( \frac{\dot{\varepsilon}}{\bar{\varepsilon}_D} \right) \right]^{1/p_i} \quad \text{(6)}$$

$$s_D(T, \dot{\varepsilon}) = \left[ 1 - \frac{kT}{g_0 \bar{\varepsilon}_D \mu b^3} \ln \left( \frac{\dot{\varepsilon}}{\bar{\varepsilon}_D} \right) \right]^{1/p_D} \quad \text{(7)}$$

where $g_0$, $\bar{\varepsilon}_D$, $p$ and $q$ are constants and their suffixes I and D refer to yielding and work hardening, respectively; $b$ is the magnitude of Burgers vector. The threshold stress $\hat{\sigma}_D$ increases with strain by accumulation and annihilation of dislocations, which is connected with the work hardening law proposed by Kocks,\textsuperscript{24)}

$$\hat{\sigma}_D = \hat{\sigma}_{DS} \left[ 1 - \exp \left( -\frac{-\Theta_0 \dot{\varepsilon}}{\hat{\sigma}_{DS}} \right) \right] \quad \text{(8)}$$

Here, $\Theta_0$ means the stage II work hardening rate and $\hat{\sigma}_{DS}$ the saturation stress of $\hat{\sigma}_D$ at an arbitrary temperature and strain rate. Thus, $\hat{\sigma}_{DS}$ is associated with the saturated dislocation substructure obtained by extremely severe plastic deformation and hence dependent on deformation condition, which does not appear in tensile deformation because necking starts before reaching such a situation. This is the outline of the model and several applications to real engineering steels have been reported elsewhere.\textsuperscript{26–28)}

Fig. 12. Effect of grain size on flow curves of the FC steels obtained by tension tests with various strain rates.

Nominal stress–strain curves of ferrite-cementite (FC) steels with various grain sizes obtained with three strain rates are summarized in Fig. 12.\textsuperscript{10} The stress at the lower yield point (LYS) and the flow stress increase with a decrease in grain size at the strain rates of $3.3 \times 10^{-4}$ s$^{-1}$, $10^3$ s$^{-1}$, and $10^9$ s$^{-1}$. It has already been reported that the effect of ferrite grain size on flow stress can be mainly ascribed to an increase in the athermal stress.\textsuperscript{13,30,31)} Lüders strain becomes larger with a decrease in grain size.\textsuperscript{30,31)} Figure 13 shows the change in 7% and 12% flow stresses with ferrite grain size as a function of strain rate. As seen, little strain rate dependence is observed. This means the second and third terms of the Eq. (5) are scarcely affected by grain size and hence grain refinement strengthening mainly contributes to the athermal stress. This is consistent with the interpretation obtained from compression test data using tiny specimens by Jia et al.\textsuperscript{33)} The calculations by Eq. (5) for the FC steels by taking the grain size effect into the athermal stress were reported, where the parameters for the Kocks–Mecking model are determined from the results obtained by conventional tension test.\textsuperscript{34)} Wei et al. have reported the strain rate sensitivity in ultrafine grained alloys is large in fcc alloys but very small in bcc alloys.\textsuperscript{35)} When the measured flow stresses and strain rate are plotted in a log–log diagram, the slope of the line, i.e., the conventional strain sensitivity parameter ($m$-value), becomes 0.023 for 13.6 $\mu$m and 0.0108 for 0.47 $\mu$m indicating very small grain size dependence. It may stem from the interaction between dislocation and grain boundaries, presumably from the absorption and emission of dislocations.
5. Deformation Mechanism of Ultrafine Grained Steels

The drawing is a traditional deformation process which can give giant strain to produce ultra-high strength. True strain of approximately 4 was commonly given to ferrite and pearlite steels with different carbon concentrations in order to obtain finer microstructure. Figure 14 shows the effect of carbon concentration on tensile strength of the drawn steel wires. As seen, tensile strength increases linearly with increasing of carbon concentration. According to TEM observations, grain size (lamellar spacing) becomes finer with increasing of carbon concentration. Two typical cases are presented in Fig. 15. In case of a low carbon steel wire, grain size is approximately 100 nm and many dislocations are visible. On the other hand, in a 0.8C pearlite steel wire, grain size is much smaller, approximately 20 nm, dislocation image is difficult to be confirmed and cementite is hardly observed. The structures are too fine and too complicated to obtain quantitative microstructural data. Then, we employed a profile analysis for neutron diffraction data obtained from a bundle of wires and the results are shown in Fig. 16. As seen, grain size (diffraction mosaic size) decreases with an increase in strain of drawing but dislocation density (estimated from so called microstrain) shows a maximum before true strain of 4. Figure 17 shows the fractography of the 0.8C steel wire fractured at RT in which the fracture surface consists of dimple patterns indicating that the plastic mode is still slip, i.e., dislocation motion. The 0.8C steel wire was pulled under neutron diffraction and nonlinear elastic deformation behavior (it is reversible) is observed above 3.5 GPa in the applied stress versus 110 lattice strain.
The formation mode is slip, strength above 3 GPa and dimple fracture indicating the deformation. The 20 nm grain sized steel wire shows tensile strength mainly through grain refinement. The athermal stress is contributed to increasing of the strength by the Kocks–Mecking model, in which the grain refinement overcomes by introducing dispersed hard grains, particularly metastable hard austenite grains. The drawback of poor uniform elongation can be overcome by introducing dispersed hard grains, particularly metastable hard austenite grains. The strength of ultrafine grained steel wires is dependent on carbon content mainly through grain refinement and the original figure given in Ref. 42). It should be noted that such a high strength wire with grain size of 10–20 nm fractures in a ductile manner even at 77 K showing dimple pattern on the fracture surface.

5. Summary

Figure 18 summarizes the grain size effect on the yield strength diagram simplified after Ref. 42), in which the topics mentioned above are inserted. As seen, this review covers studies on grain size of 20 to 20 000 nm, where the dislocation motion is the dominant deformation mode. The followings are main conclusions to be claimed.

(1) With decreasing of grain size, work-hardening rate decreases to result in loss of uniform elongation.

(2) Lüders strain is described as a function of work hardening rate at the end of Lüders deformation.

(3) The drawback of poor uniform elongation can be overcome by introducing dispersed hard grains, particularly metastable hard austenite grains.

(4) Effect of strain rate on flow stress is well described by the Kocks–Mecking model, in which the grain refinement contributes to increasing of the athermal stress.

(5) The strength of ultrafine grained steel wires is dependent on carbon content mainly through grain refinement. The 20 nm grain sized steel wire shows tensile strength above 3 GPa and dimple fracture indicating the deformation mode is slip, i.e., dislocation motion.

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