

Relative contributions of solutes and forest dislocations to thermally activated plasticity in hydrogen-alloyed Fe-Cr-Ni austenitic steels: analyzing stress-dependence of *Haasen plot*

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ABSTRACT

A competitive relationship between solute atoms and forest dislocations in determining the kinetics of thermally activated plasticity was studied for a Type310S (Fe-24Cr-19Ni) austenitic steel containing ~8500 at ppm hydrogen (H), where solute H promotes solid solution-hardening through an increase in the effective stress. The investigation was performed by constructing *Haasen plot* (i.e., strain rate sensitivity (SRS) vs. flow stress) based on stress relaxation tests, and by analyzing its evolution in relation to the decay of effective stress. In the regime of high effective stress, alloying solute elements—whose overcoming by dislocations is retarded by the presence of H—acted as primary rate-controlling obstacles to dislocations, giving rise to a relatively large and strain-independent SRS. In terms of *Haasen plot*, these manifested as an increase in the intercept and a decrease in the slope. Beyond the phenomenological findings of these H-induced characteristic changes, the present study elaborates their physical origin and transient nature. As the effective stress decreased and dislocation density increased, such predominance of solutes was progressively taken over by forest dislocations, leading the *Haasen plot* for H-charged specimens to approach that for non-charged specimen. These new insights further establish how H, alloying elements, and forest dislocations sequentially compete as rate-controlling obstacles depending on mechanical loading condition and internal state of the material.

1. Introduction

In the study field of hydrogen (H)-metal interactions in structural materials, there has been a long-standing argument over the behavior of crystal dislocations in the plasticity of face-centered cubic (FCC) metals and alloys under the presence of solute H [1–5]. The authors have recently investigated, in our companion paper [6], room-temperature thermally activated deformation and the predominant rate-controlling obstacles to dislocation motion in a Type310S (Fe-24Cr-19Ni) austenitic steel containing ~8500 at ppm H [6]. Through rigorous analysis of stress relaxation behavior and the stress-dependence of activation volume, V (i.e., the volume associated with a dislocation segment overcoming a short-range energy barrier), two key assertions were drawn: (i) dislocation motion in austenite matrix remains primarily governed by intrinsic obstacles such as alloying elements, even in the presence of H; and (ii) H imposes an additional energy barrier, increasing the effective stress (i.e., time-/strain rate-dependent stress component) required for dislocations to bypass these intrinsic obstacles. These insights refined

the model of dynamic H-dislocation interactions proposed in our earlier studies [7,8] and deepen the understanding of how H impacts thermally activated plasticity and causes solid solution-hardening [2,7–11]. However, the role of forest dislocations, which becomes increasingly significant with strain and control the deformation kinetics in cooperation with matrix obstacles, was beyond the scope of discussion.

Dislocation motion through a field of single or multiple obstacle types entails complex processes, making the additivity of individual obstacle contributions to the total flow stress, τ , a central issue in dislocation dynamics [12–19]. Nonetheless, linear stress additivity has fortunately been established when one set of obstacles differs markedly in their strength and density to the other [16,20–22]. Such conditions are exemplified by forest dislocations (strong, coarsely spaced obstacles) combined with solute atoms (weak, densely distributed obstacles) in concentrated solid solutions [16,17,20,22]. Given the flow stress components from solute atoms and forest dislocations are respectively τ_s and τ_d , one yields $\tau = \tau_s + \tau_d$. The latter component progressively increases with an increase in strain (i.e., strain-hardening), while the former is

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often assumed as strain-independent and primarily exhibits a time- and strain rate-sensitive (*i.e.*, effective stress) character. Owing to this simple law, the additivity also holds for their differentiations as [22,23]:

$$\frac{\partial \tau}{\partial \ln \dot{\gamma}} = \frac{\partial \tau_s}{\partial \ln \dot{\gamma}} + \frac{\partial \tau_d}{\partial \ln \dot{\gamma}} \quad (1)$$

This parameter is called strain rate sensitivity (SRS) of flow stress, inversely proportional to V [22,24]:

$$\frac{\partial \tau}{\partial \ln \dot{\gamma}} = \frac{kT}{V} \quad (2)$$

For the derivation of Eq. (2), the readers shall refer to [24]. Through the use of Eq. (1), we can somehow distinguish the influences of solutes-dislocation and dislocation-dislocation interactions on the kinetics of thermally activated deformation.

Given that τ_s is an inherent glide resistance of the matrix and approximated by yield stress, τ_y , Eq. (1) becomes:

$$\begin{aligned} \frac{\partial \tau}{\partial \ln \dot{\gamma}} &= \tau \frac{\partial \ln \tau}{\partial \ln \dot{\gamma}} = \tau_y m + \tau_d \frac{\partial \ln \tau_d}{\partial \ln \dot{\gamma}} = \left(\frac{\partial \ln \tau_y}{\partial \ln \dot{\gamma}} - \frac{\partial \ln \tau_d}{\partial \ln \dot{\gamma}} \right) \tau_y + \frac{\partial \ln \tau_d}{\partial \ln \dot{\gamma}} \tau \\ &= (m_y - m_d) \tau_y + m_d \tau \end{aligned} \quad (3)$$

Here, m is relative rate sensitivity, generally assumed inherent for a given obstacle type, and the subscripts y and d denote its solutes and forest dislocation components, respectively. Thus, if one measures SRS at various strains and plots them as a function of τ , the slope corresponds to m_d , while the intercept reflects the contribution from m_y [23]. This procedure is called *Haasen plot* analysis, applied to the investigations of strengthening mechanisms in a variety of structural materials [21, 25–29]. In pure FCC metals where τ_y is very small, the *Haasen plot* becomes a straight line passing close to the origin [21,25,30]. Meanwhile, positive intercepts were often identified in FCC solid solution alloys owing to the contributions of solutes to τ_y [21,23,25].

The authors have employed *Haasen plot* analysis to the H-charged Type310S austenitic steel, identifying two remarkable H-impacts on the shape of such a plot [8]: (i) increase in intercept; and (ii) decrease in slope. The effect on intercept was the backbone of our assertion that H provides thermally activatable energy barrier against dislocation motion. On the other hand, the change in slope (*i.e.*, the change in forest dislocation contribution) has not yet well been interpreted. Although the contributions of solutes and forest dislocations to the flow stress are additive, their respective glide activation energies, ΔG_s and ΔG_d , are not. Instead, they compete with each other, and one predominates in controlling the deformation kinetics [17,18,31]. Critically, their predominance is governed by the applied stress, with weaker obstacles becoming increasingly influential with the rise in stress level as first pointed out by Schoeck [31]. This competition arises from the differences in activation volumes and zero-stress activation energies between solutes and forest dislocations; besides, the activation volume itself is stress-dependent [6,22,32,33]. *Haasen plot* is typically constructed through a measurement of stress-strain curve under a constant strain rate, accompanied by test interruptions to evaluate mechanical transients [8,21,27,29,34]. However, this approach provides only limited information focusing merely on a specific deformation condition. Thus, it is now desired to evaluate the stress-dependence of *Haasen plot*, alongside that of the activation volume [6]. Such an analysis would enable a more perfect comprehension about how solute H, alloying elements, and forest dislocations under a given applied stress competitively behave among these multiple obstacle-types and impacts the *Haasen plot* shape.

This study applies the above approach to our previously obtained V -stress relationship data [6], measured along the relatively long-term relaxation curves at various strains. These data for Type310S steel were converted into SRS via Eq. (2), followed by the construction of *Haasen plot* at some selected stress levels. Ultimately, our model of

thermally activated dislocation motion involving H is further refined into the form that can successfully account for the H-induced change in *Haasen plot* slope. It further establishes how H, alloying elements, and forest dislocations sequentially compete as rate-controlling obstacles depending on mechanical loading condition and internal state of the material, establishing a more complete picture of thermally activated deformation in H-charged austenitic steel at ambient temperature.

2. Methodology

2.1. Material and specimen

The material used was a solution-treated $\phi 20$ -mm rod of AISI Type310S steel with its details (*e.g.*, chemical composition, heat treatment, and initial microstructure) were provided in our foregoing papers [6,7]. Round-bar tensile specimens with 4 mm in diameter, 30 mm in gauge length, and M8 screw in the gripping part were prepared, then uniformly H pre-charged in pressurized H_2 gas at 543 K for 200 h. The H-charging at 10 and 100 MPa H_2 gas resulted in H concentrations of 2400 at ppm (44 mass ppm) and 8500 at ppm (156 mass ppm), respectively, as measured by thermal desorption analysis, while the concentration was ~ 200 at ppm (3.3 mass ppm) in the non-charged specimen [6].

2.2. Mechanical tests

Tensile tests with and without interruptions by intermittent stress relaxation and unloading have been carried out in our companion study [6], the data from which was used in the present analyses. The specimens were installed into an electromechanical test frame, Shimadzu AGX-V2, with a 20 kN load cell, then deformed at a constant crosshead speed of 0.003 mm/s (initial strain rate of 10^{-4} /s) at 295 ± 2 K. As measured by the load-crosshead displacement data during the elastic deformation of the tensile samples, the stiffness of specimen-machine assembly was ≈ 15 kN/mm.

When a predetermined strain level was reached, machine crosshead was held for 1000 s, followed by unloading down to the load of 0.2 kN and subsequent reloading toward the next targeted strain. A slower crosshead speed—0.0015 mm/s—was employed during the unloading for a more precise recording of stress-strain response. To measure strain, a contact-type strain gauge extensometer with a gauge length of 25 mm was mounted on the specimen's gauge portion. Two specimens were subjected to the tests for each H concentration. Stress relaxation and unloading procedures were implemented at nominal strains of 2, 6, 10, 14, 18, 22, 26, 30, and 34% in one specimen, whereas these were 4, 8, 12, 16, 20, 24, 28, and 32% in the other. Throughout this paper, experimentally measured normal stress-strain are designated by the symbols σ - ϵ , while their shear counterparts are denoted by τ - γ . Their conversion was done by an average Taylor factor, $M = 3.06$, for FCC polycrystals.

Besides the classification by obstacle types (Section 1), the flow stress contribution from forest dislocations, τ_d (or σ_d), comprises both isotropic and kinematic hardening components [35–37]. The former is attributed to latent strain-hardening, truly reflecting the influence of dislocation-dislocation interactions on thermally activated deformation [21,30,38]. The latter part corresponds to polarized internal stress (*i.e.*, time-/strain rate-insensitive stress component)—commonly called back stress, τ_{Back} (or σ_{Back}) [35–37,39]—caused by heterogeneous dislocation structures or other microstructural heterogeneities such as deformation twins [36,39–41]. Since σ_{Back} is also involved in the total flow stress, τ (*i.e.*, horizontal axis of *Haasen plot*), its evolution with strain may affect the shape of *Haasen plot* as well. To exclude this secondary effect, σ_{Back} in non- and H-charged specimens were measured by the stress-strain hysteresis method proposed by Cottrell [42] and later refined by Handfield and Dickson [43]. In essence, the analysis utilizes backward plastic flow and the corresponding Bauschinger strain during unloading [37].

Further procedural details are provided in Appendix A1.

2.3. Characterization of deformation microstructure

To connect flow behaviors to deformation substructures particularly at large strains where deformation modes other than dislocations (e.g., deformation twins [44,45]) possibly intervene, we applied electron microscopies to the specimens deformed to 0.30 true strain. The specimen's mid-diameter section was cut along tensile axis and polished by colloidal SiO_2 suspension. Crystallographic analysis was performed by electron backscattering diffraction (EBSD) in a field-emission scanning electron microscope (SEM), Zeiss Sigma, equipped with Bruker QUAN-TAX system operated at 20 kV.

Disks with a diameter of 3 mm and a thickness of $\approx 100 \mu\text{m}$ were also prepared from the deformed specimens so that the disk plane becomes perpendicular to the tensile axis. The disk centers were thinned by twin-jet electrochemical polishing in Struers TenuPol 4 using an electrolyte of 10% perchloric acid and 90% ethanol. Deformation microstructures in the thinned parts were observed by a transmission electron microscope (TEM), JEOL JEM-2100, at an acceleration voltage of 200 kV.

3. Results

3.1. Plastic flow behavior

Fig. 1(a) presents the true stress–strain and strain-hardening rate curves for non-charged and H-charged specimens under monotonic tensile loading. These stress–strain curves are compared with corresponding results from the tests involving crosshead-holding and unloading in Fig. 2. In the early to intermediate deformation stages, 2400 and 8500 at ppm H increased the flow stress by 10–20 MPa and ≈ 50 MPa, respectively, producing nearly parallel upward shifts in the stress–strain curves (cf. the inset in Fig. 1(a)). Up to a strain of 0.2, H had little effect on the strain-hardening rate except for the initially low hardening rate in the specimen containing 8500 at ppm H [9,46]. Beyond this, however, the curves for H-charged specimens deviated upward from parallelism, with progressively enhanced strain-hardening rates compared to the non-charged condition—shaded domain in Fig. 1. Excluding the crosshead-holding and unloading segments, the stress–strain responses in monotonic and relaxation-involved tests were identical for a given H concentration (Fig. 2), indicating that transient stress

relaxation and unloading did not have impact on subsequent deformation behavior. For further details of the coincidence between the stress–strain responses in these two test types, the readers shall refer to Appendix A2.

3.2. Stress decomposition

Fig. 3(a)–(c) magnifies representative stress–strain responses during unloading and reloading near true strain of 0.06, 0.13, and 0.29 for both non- and H-charged specimens. For simplicity, the data of the specimen containing 2400 at ppm H were omitted. The specimens initially unloaded elastically, followed by reverse yielding well before the stress reached minimum, resulting in distinct hysteresis loops whose width gradually increased with strain. This increase in hysteresis loop width is a consequence of an escalation in the Bauschinger strain, $\Delta\epsilon_B$, although the influence of solute H on the reverse yielding behavior was obscure only from Fig. 3.

Fig. 1(b) shows the evolution of σ_{Back} , determined from the detailed analysis of unloading curves (Fig. 3) using the method described in Appendix A1 [43], along with the total flow stress (i.e., externally applied stress), σ . Up to a true strain of ~ 0.20 , σ_{Back} remained largely unaffected by H-charging, supporting our prior statement that the increased flow stress by H primarily stems from an increase in effective stress [7]. At higher strains, however, σ_{Back} in H-charged specimens slightly exceeded that in the non-charged one, with a maximum difference of ≈ 50 MPa at a strain of ≈ 0.3 . This H-induced enhancement in σ_{Back} accounts for the increased strain-hardening rate at a true strain beyond 0.20 (Fig. 1(a)).

3.3. Stress relaxation behavior

In Fig. 4, stress relaxation curves during the crosshead-holding at some representative strain levels are presented in the form of logarithmic stress reduction rate, $\ln(-\dot{\sigma})$, vs. stress decay, $\Delta\sigma$ (i.e., the magnitude of stress drop from the beginning of relaxation). For more general expression of these relaxation curves in “ $\Delta\sigma$ vs. time” plots, the readers shall refer to our previous paper [6]. Beyond the crosshead-holding time over 500 s, stress relaxation became too slow to reliably quantify the stress decaying rate. Accordingly, the following analysis focuses on data within the first 400–500 s.

During stress relaxation, the elastic strain stored in the whole

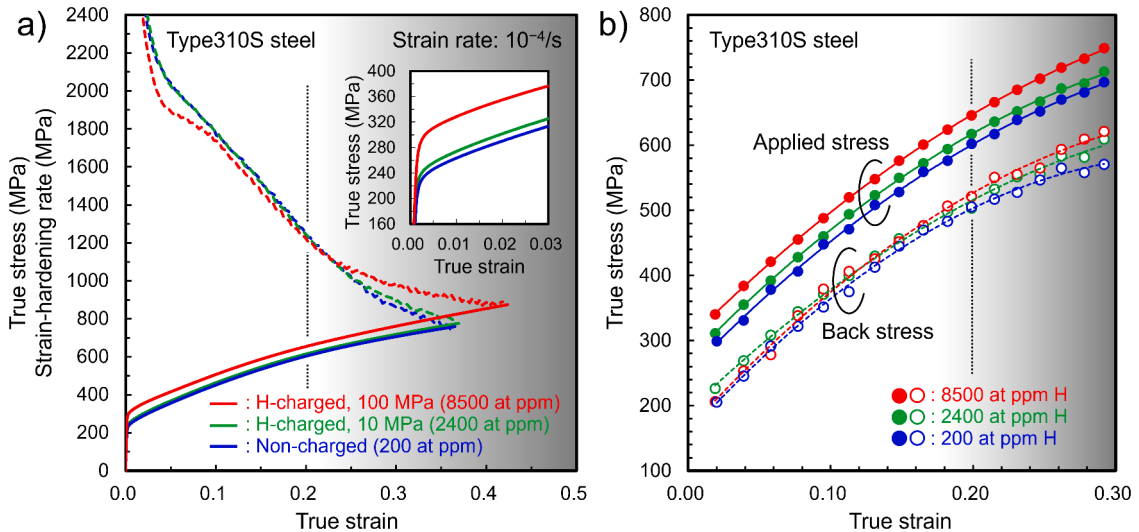


Fig. 1. (a) True stress–strain (solid lines) and strain-hardening rate (dotted lines) curves of the Type310S steel with three different H concentrations at 295 K (reproduced from [6]). The inset in (a) magnifies the behavior around yield stress. (b) shows the evolution of back stress, σ_{Back} (see Section 2.2), as a function of strain, together with the corresponding applied stress. The dotted vertical lines in each diagram denote the border at which σ_{Back} in H-charged specimens started to overwhelm that in non-charged one.

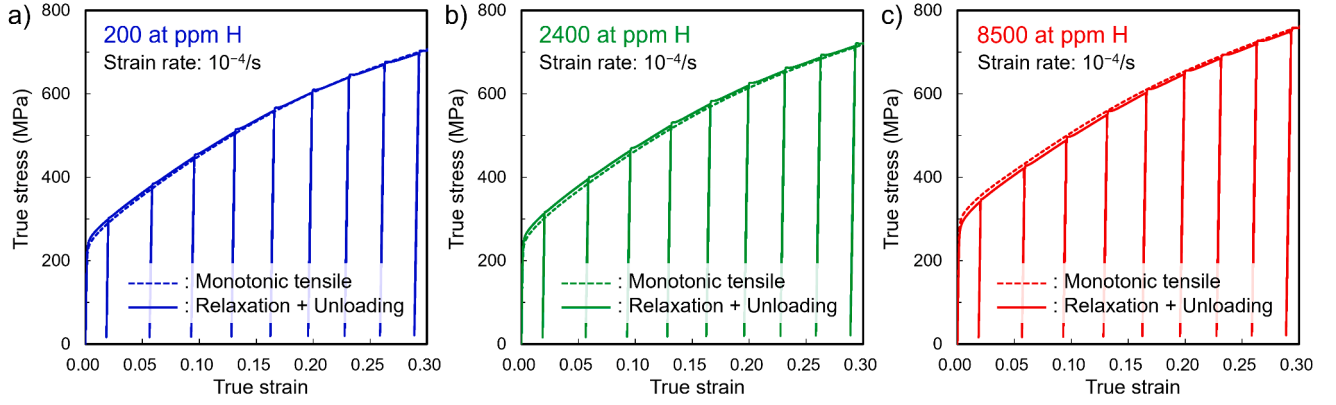


Fig. 2. Comparison of the true stress-strain responses in monotonic tensile tests (dashed lines) and the tests involving intermediate crosshead-holding and unloading (solid lines) under (a) 200 at ppm H, (b) 2400 at ppm H, and (c) 8500 at ppm H. Regarding the latter type of tests, only the data from one specimen out of the two-specimen set for each H-concentration condition are plotted for better visibility.

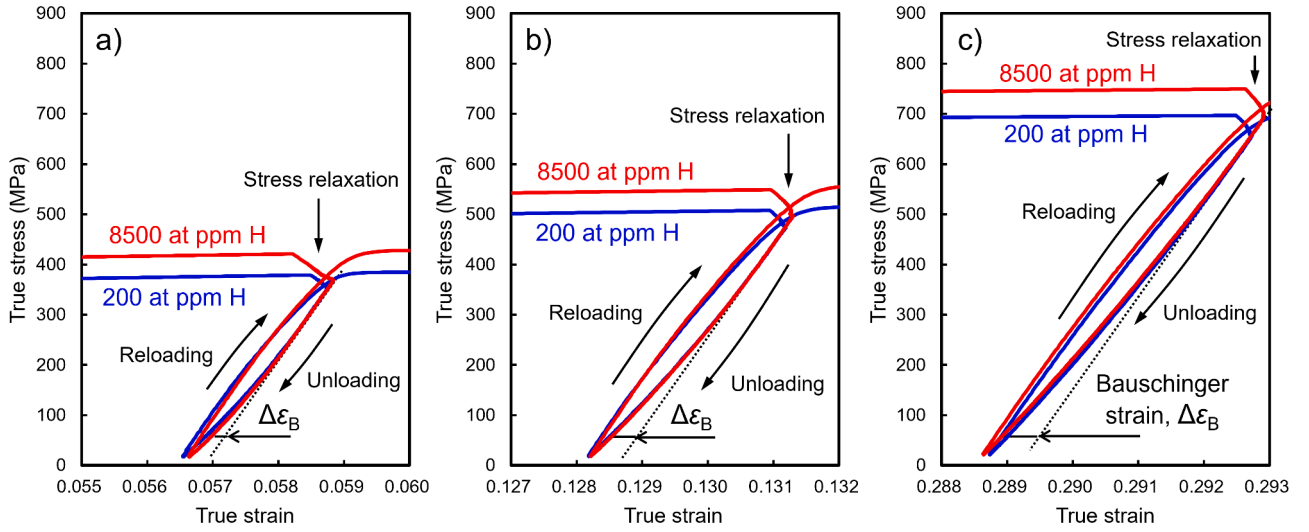


Fig. 3. Reverse and forward yielding behaviors during unloading and subsequent reloading in specimens containing 200 and 8500 at ppm H at three different strain levels (a)–(c). The curves describe clear hysteresis loops owing to the Bauschinger strain.

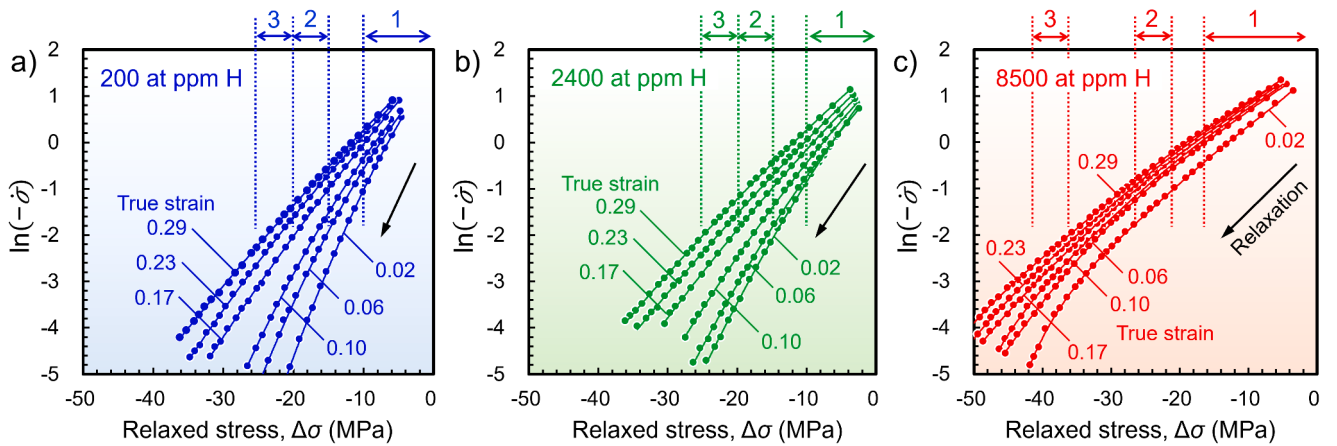


Fig. 4. Stress relaxation curves in (a) 200, (b) 2400, and (c) 8500 at ppm H containing specimens subjected to various strain levels. The curves are shown in the form of $\ln(-\dot{\sigma})$ vs. $\Delta\sigma$, the slope of which yields the corresponding activation volume, V (cf. Eq. (5)). The black arrows in each diagram denote the direction of the progress of stress relaxation. Only the data from one specimen out of the two-specimen set for each H-concentration condition are plotted for better visibility.

specimen-machine assembly is progressively replaced by the plastic strain within the specimen's gauge part. Thus, the plastic strain rate during the relaxation, $\dot{\epsilon}$, can be expressed as:

$$\dot{\epsilon} = -\dot{\sigma}/E \quad (4)$$

where E is the elastic stiffness of the specimen-machine system. Inserting Eq. (4) into Eq. (2) and considering shear-to-normal stress-strain conversion, one yields:

$$V = kT \frac{\partial \ln(M\dot{\epsilon})}{\partial \sigma/M} = MkT \frac{\partial \ln(-M\dot{\sigma}/E)}{\partial \sigma} = MkT \frac{\partial \ln(-\dot{\sigma})}{\partial \sigma} \quad (5)$$

Eq. (5) demonstrates that calculating the local slope of $\ln(-\dot{\sigma})-\Delta\sigma$ plots in Fig. 4 provides the activation volume, V , at any moment during the relaxation period. Note that the V defined in Eq. (5) should more appropriately be regarded as an “apparent activation volume” because it includes the influence of strain-hardening associated with plastic strain accumulated during the relaxation process (i.e., it is slightly larger than the “true activation volume” free from this effect). Nevertheless, our previous study showed that solute H affects both quantities in the same manner [8], supporting the validity of the present approach in using the former as an indicator of material behavior under different H concentrations.

Fig. 4 reveals downward curvatures (i.e., increasing slope) of such $\ln(-\dot{\sigma})-\Delta\sigma$ curves as the stress decreases with the elapse of time (arrows in Fig. 4), the significance of which was larger in the specimens containing higher H concentration. The presence of this downward curvature indicates the stress-dependent character of V —increases as stress decreases—and, in turn, the stress-dependence of SRS (cf. Eq. (2)).

3.4. Stress-dependence of Haasen plot

On the basis of $\ln(-\dot{\sigma})-\Delta\sigma$ curves and Eq. (5), V values were derived by selecting some regimes from each curve based on stress levels and deriving their local slopes. The stress ranges used for this analysis are highlighted at the top of Fig. 4 with the numbers 1, 2, and 3. Considering the linear additivity of flow stress (i.e., $\tau = \tau_s$ (or τ_y) + τ_d) wherein strengthening contributions of alloying elements and H (i.e., effective

stress component related to these solutes) are assumed to be nearly constant with strain, this regime-wise approach captures how the Haasen plot evolves with a given amount of decrease in effective stress throughout the stress-strain curve. Physically, the method is equivalent to measure V by starting the relaxation after a slower prior strain rate (i.e., $<10^{-4}$ /s), considering that the plastic strain rate also decays progressively with the relaxation stress drop [8,46]. Fig. 5(a) shows the V for every stress regime in Fig. 4, plotted against strain. The corresponding Haasen plots were constructed by converting these V into SRS using Eq. (2). As the horizontal axis of Haasen plots, the flow stress at the onset of each relaxation was used for Regimes 1 in Fig. 4, while the drop from such initial value due to relaxation was accounted for Regimes 2 and 3. Note that the choice of those stress regimes is essentially arbitrary as long as the following two requirements are satisfied: (i) the selected portion corresponds to the same amount of stress relaxation at all strain levels, and (ii) the selected stress range is large enough to determine the slope of $\ln(-\dot{\sigma})-\Delta\sigma$ response with sufficient accuracy. Among the various possibilities examined by the authors, the Regimes 1–3 were chosen because they make the stress-dependence of Haasen plot most clearly visible in Fig. 5(b).

As shown in Fig. 5(b), the resultant Haasen plots all exhibited positive intercepts and linearly increasing fashion with respect to stress, verifying the constant m_y and m_d in Eq. (3) under a given level of effective stress. Focusing on the Regimes 1–initial relaxation stage—in non- and H-charged specimens, increasing H concentration triggered two prominent changes in Haasen plot: (i) an increase in intercept and (ii) a decrease in slope. In the non-charged specimen, the reduction in stress level toward Regimes 2 and 3 caused a minute increase in Haasen plot slope, as well as a slight downward shift of whole the plot line. Then, the intercept in Regime 2 passes close to the origin as for the case of pure FCC metals [21,25,47], besides the intercept became even negative in Regime 3. Such a negative intercept may reflect athermal contributions of long-range barriers (e.g., grain boundaries) to the flow stress [21], becoming increasingly predominant as the effective stress decays in the end phase of relaxation. By contrast, although these stress-dependent trends were the same in H-charged specimens, it appeared more remarkably as the concentration of H increased. Eventually, the plot

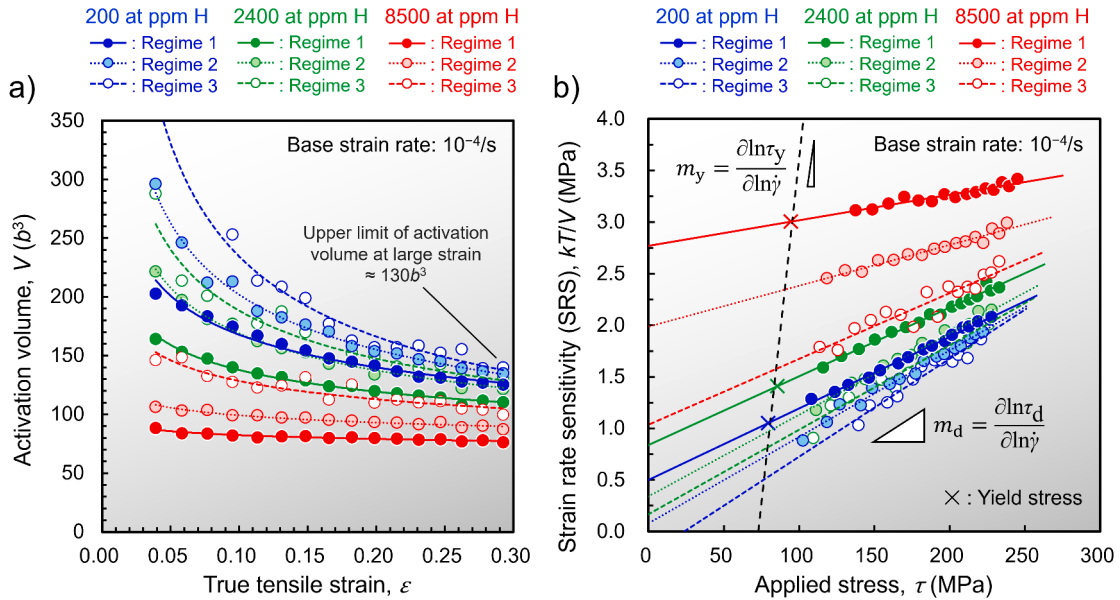


Fig. 5. (a) Transition of activation volume, V , as a function of true strain. The Regimes 1–3 under each H concentration correspond to the portions of relaxation curves marked in Fig. 4. These V values in (a) were converted into strain rate sensitivity (SRS), kT/V , via Eq. (2) and reproduced in (b) as Haasen plot (i.e., SRS versus flow stress). The yield (0.2% proof) stress, σ_y , under each H concentration was converted into its shear counterpart, τ_y , and plotted by cross mark in (b). The graphical description of the parameters m_y and m_d , defined in Eq. (3), are also shown in (b).

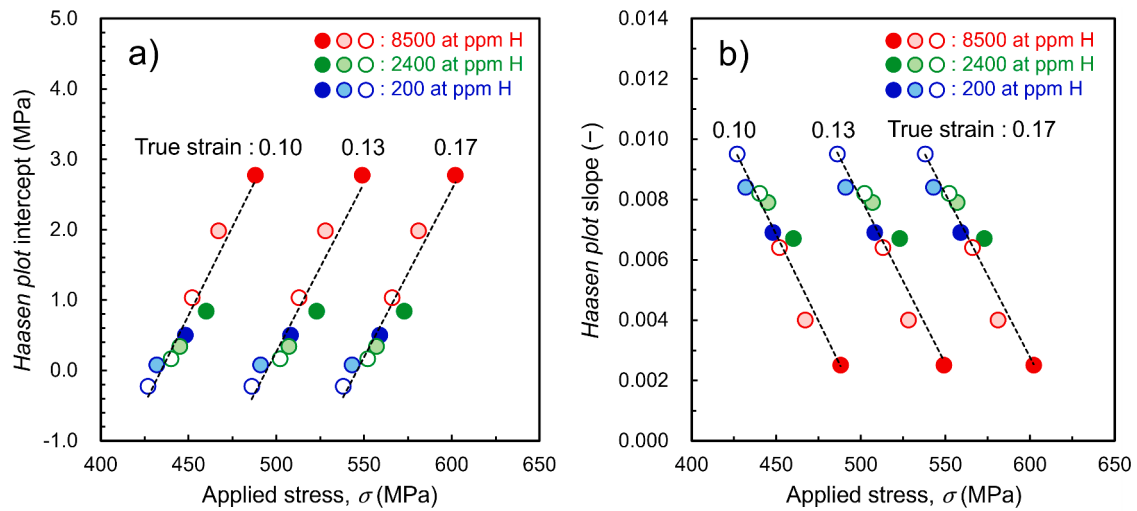


Fig. 6. (a) Intercept and (b) slope of the *Haasen plot* shown in Fig. 5(b) as a function of applied stress at some representative strain levels. The color and style of each plot mark correspond to the legends in Fig. 5.

lines of H-charged specimens tended to merge into that of the non-charged case, diminishing the above two H-induced characteristics (i) and (ii). In Fig. 6, the *Haasen plot* intercepts and slopes of the nine data-sets in Fig. 5(b) are reproduced as a function of applied stress, σ , at three representative strain levels. Notably, all the plot points lay close to

a single linear line, inferring that these two parameters follow an identical effective stress-dependence irrespective of the presence and absence of H.

Returning to Fig. 5(a), the positive slope of the *Haasen plots* in Fig. 5 (b) corresponds to a parabolic decrease in activation volume, V , with

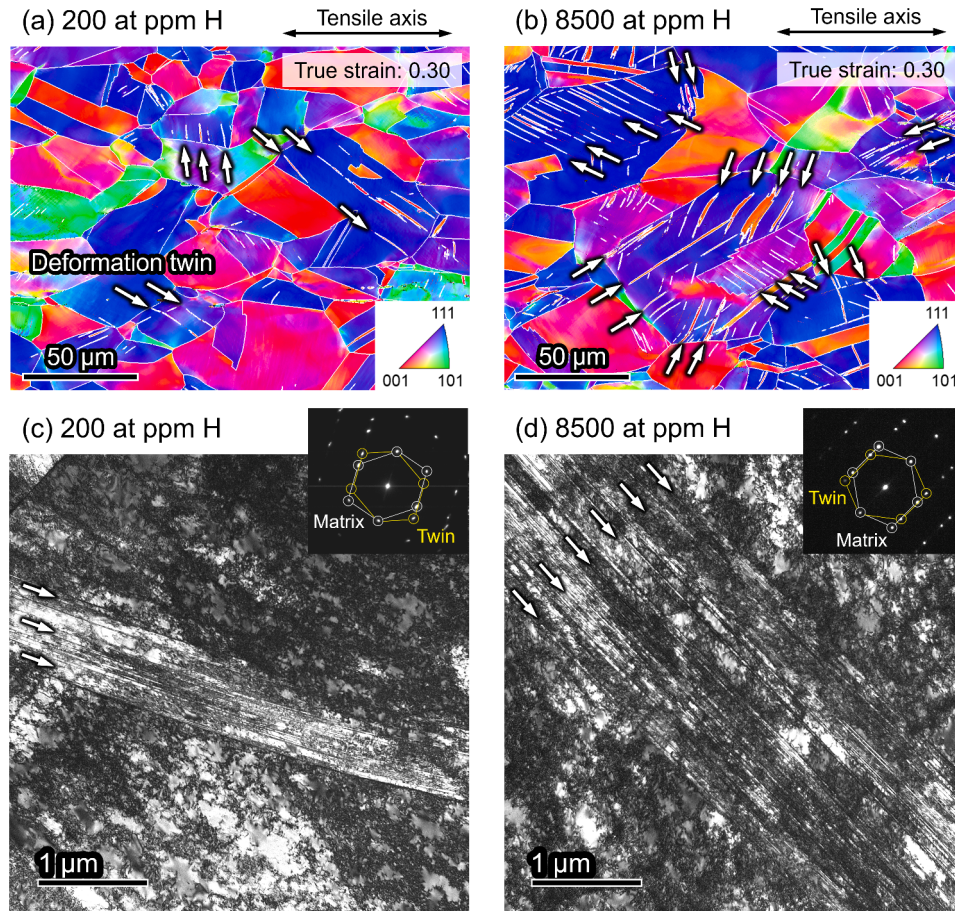


Fig. 7. Deformation microstructures in (a)(c) non-charged (200 at ppm H) and (b)(d) H-charged (8500 at ppm H) specimens deformed to 0.30 true strain characterized by (a)(b) EBSD (beam step size: 120 nm) and (c)(d) TEM. The white arrows in each micrograph denote deformation twins. The color code in (a) and (b) denotes the crystallographic orientation along the tensile axis. The tensile axes of the grains captured in (c) and (d) are close to $\langle 111 \rangle$, which are nearly perpendicular to the images.

increasing strain. The H-induced effects (i) and (ii) caused an overall reduction and weakened strain-dependence of V in Regime 1, whereas these two features gradually diminished as the stress decreased into Regimes 2 and 3. In the non-charged specimen, V exceeded $200b^3$ at strains below 0.1 and asymptotically decreased to $120\text{--}140b^3$ at a strain of 0.3.

3.5. Deformation microstructure

Fig. 7(a) and (b) show EBSD inverse pole figure (IPF) maps of the deformation microstructures in non- (200 at ppm H) and H-charged (8500 at ppm H) specimens deformed to 0.30 true strain, captured by a beam step size of 120 nm. The color coding indicates crystallographic orientations along the tensile axis (TA). Due to large deformation, grains rotated toward $\langle 001 \rangle // \text{TA}$ and $\langle 111 \rangle // \text{TA}$ directions, with prominent deformation twin bands appearing in $\langle 111 \rangle // \text{TA}$ -oriented grains, as marked by arrows. H-charging markedly increased the density of deformation twins.

Fig. 7(c) and (d) present TEM images of the substructures existing with deformation twins in the grains oriented close to $\langle 111 \rangle // \text{TA}$. Each twin band observed in the IPF maps (Fig. 7(a) and (b)) practically comprises a bundle of nanoscale twin plates (tens to hundreds of nanometers thick), surrounded by a high density of dislocations forming well-defined cell structures with the diameters of 200–500 nm. These cell sizes were significantly smaller than the inter-twin bundle spacing of few to tens of micrometers in Fig. 7(a) and (b). The twin bundles traversed the dislocation cells, suggesting that cell formation preceded twin evolution [48,49]. Apart from the enhanced deformation twin density, H had negligible effects on other microstructural features such as cell size.

4. Discussion

The present experiments revealed two major H-effects on *Haasen plot*: (i) an increased intercept and (ii) a decreased slope (Fig. 5(b)), accompanied by an overall enhancement in SRS and corresponding reduction in activation volume, V (Fig. 5(a)). These trends closely reproduced our previous observations from short-term (30 s) stress relaxation tests [8]. However, all these effects diminished with decreasing stress, underscoring that constructing the *Haasen plot* solely at the flow stress level under a fixed strain rate is insufficient to fully capture H-induced changes in dislocation kinetics across diverse obstacle types, as noted in Section 1. This newly found transient nature of *Haasen plot* emphasizes the sequential competition between H, alloying elements, and forest dislocations as rate-controlling obstacles depending on mechanical loading condition and internal state of the material.

4.1. Screening the back stress-effects

An essential premise for the discussions above and below is the invariance of internal or back stress in non- and H-charged specimens in beginning-to-intermediate deformation stages (Fig. 1(b)). This fact is consistent with our former results [7] where internal/back stress in the same steel was measured by a different technique using successive stress-dip [50]. Such an invariance indicates that the back stress contribution to the stress axis of *Haasen plot* is not largely affected by H, allowing H-induced changes in the *Haasen plot* to be interpreted consistently through the effective stress framework. Although a slight increase in back stress—up to ≈ 50 MPa in normal stress (≈ 15 MPa in shear stress)—was observed in H-charged specimens at true strain exceeding 0.2 (Fig. 1(b)), its influence appears minimal, as the *Haasen plot* maintains its linearity even in the high-stress/strain regime (Fig. 5(b)).

The elevated back stress and enhanced strain-hardening rate in later deformation stage (Fig. 1(a)) are likely due to deformation twinning promoted by H (Fig. 7) [45,51,52]. While the presence of twin

boundaries could somewhat influence thermally activated deformation in FCC metals [53–55], such effect was not evident in the present Type310S steel (Fig. 5). This may be due to the much finer dislocation cell structures (200–500 nm diameters) compared to the inter-band spacing (few to tens of micrometers) of deformation twins (Fig. 7). That is, mobile dislocations interact more frequently with forest dislocations in cell walls [56–58] rather than with twin boundaries, rendering the latter interaction statistically insignificant.

4.2. Hydrogen-effects on Haasen plot

4.2.1. Increase in intercept

In light of Eq. (3), the observed increase in *Haasen plot* intercept $-(m_y - m_d)\tau_y$ suggests two possible roles of H: (I) H atoms themselves act as newly involved obstacles, that possess more rate sensitive (i.e., more thermally activatable) character than other inherent obstacles contained in the material [7,8]; (II) instead, H solely renders those inherent obstacles more rate sensitive. Our past analysis of the stress-dependence of activation volume revealed a unified V -stress relationship for both non- and H-charged specimens [6], exclusively supporting the latter scenario (II). If experimental V in the H-charged specimen probed H—an obstacle type different from inherent ones—as anticipated in the case of scenario (I), such a consistency of V -stress trend with the non-charged sample would not hold. Given that the parameter m_y reflects the matrix frictional resistance under a generally negligible Peierls barrier in FCC lattice, the responsible obstacles are alloying elements including Cr, Ni, Mn, and Si etc.

Fig. 8 schematically illustrates, in the simplest way, how H alters the strain rate sensitivity of an inherent obstacle on its force-distance profile [6]. Whatever its details, each obstacle can be represented by a hill-like energy barrier with finite slope on its mountainside, whose shape remains unaffected by H (Fig. 8(a-1) and (b-1)). Rather, H atoms segregating into the mobile dislocation [59–61] virtually superimpose an additional energy barrier, ΔG_H , atop the original obstacle profile (Fig. 8(a-1) and (b-1)) [6]. Based on the reported H diffusivity and attractive H–dislocation interaction in Fe–Cr–Ni austenite, our previous study [46] estimated that, at ambient temperature and a strain rate of $10^{-4}/\text{s}$, highly mobile H atoms tend to form an atmosphere around a dislocation, and that this H atmosphere can migrate cooperatively with the moving dislocation while remaining nearly in thermal equilibrium. Thus, the relevant additional barrier may be the energy required for these segregated H atoms to follow the glide of the dislocation core via atomic diffusion jumps, as assumed in the *diffusion-controlled glide model* described by Caillard and Martin [24]. For further details of our model for dynamic H–dislocation interaction in the background field of alloying elements, readers are referred to Ref [6].

In the absence of H-effect— ΔG_H , the total activation energy to surmount this potential hill, ΔG_0 , is partitioned into the thermal activation (Gibbs free) energy supplied by atomic vibration, ΔG , and the mechanical work term, τ^*V (Fig. 8(a-1) and (b-1)) [22,24,62,63]:

$$\Delta G = \Delta G_0 - \tau^*V \quad (6)$$

Under given ΔG and effective shear stress, τ^* , the plastic shear strain rate, $\dot{\gamma}$, is then expressed using an Arrhenius type rate equation as [22, 24,62,63]:

$$\dot{\gamma} = \dot{\gamma}_0 \exp\left(-\frac{\Delta G}{kT}\right) \quad (7)$$

where the pre-exponential factor, $\dot{\gamma}_0$, includes the mobile dislocation density, average obstacle spacing, and vibrational frequency of dislocation segment [22,24]. Eq. (7) defines that, under given material state, strain rate, and temperature, ΔG is probabilistically constant. Because such a fixed statistical average of ΔG is partially consumed to compensate for ΔG_H , τ^* must be increased to allow the H-charged specimen to conform with externally imposed strain rate (areas hatched with red and

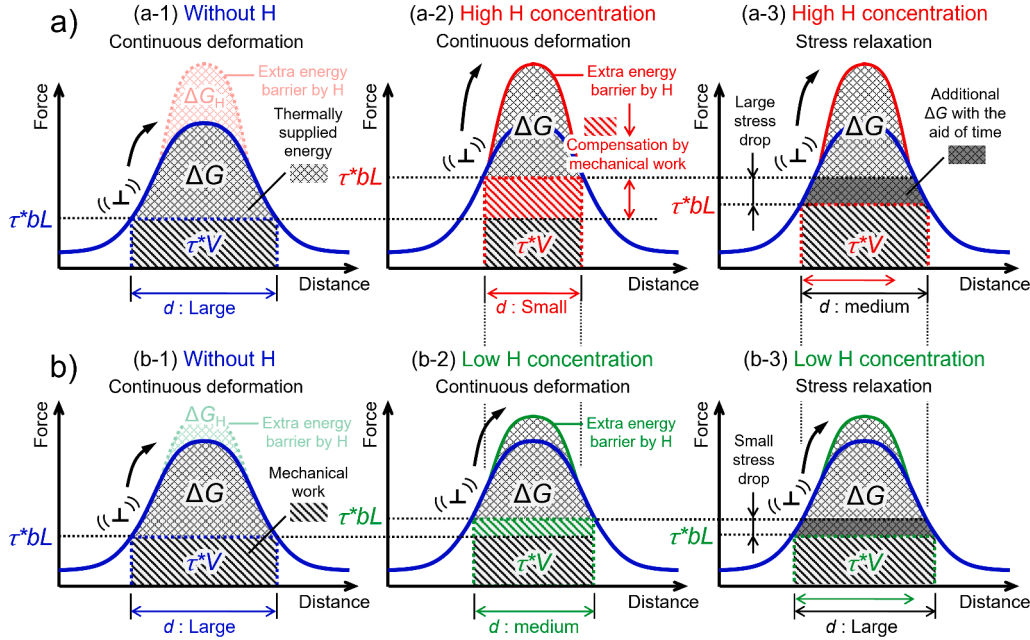


Fig. 8. Schematic illustrations of how H and stress level affect the kinetics of dislocation glide through the obstacles inherently contained in the material. (a) and (b) compare the non-charged state with (a) high and (b) low H concentration conditions, respectively. (a-1), (a-2), (b-1), and (b-2) describe the situation in monotonic tensile deformation with a fixed strain rate, while (a-3) and (b-3) denote the transition into stress relaxation phase.

green in Fig. 8 (a-2) and (b-2)). This rise in effective stress accounts for the elevated flow stress—solid solution-hardening with upward shifts of stress-strain curves (Fig. 1(a))—in H-charged specimens compared to non-charged ones in continuous deformation under an identical strain rate condition.

Activation volume, V —the inverse of SRS (Eq. (2))—is geometrically regarded as a product of the effective obstacle width/extent, d (Fig. 8), the length of activated dislocation segment, L , and the Burgers vector, b [22,24]. As evident from Fig. 8 (a-1) versus Fig. 8 (a-2), an increase in τ^* along a finite obstacle profile slope necessarily reduces d . Additionally, the solute-dislocation interaction model employed in our previous analysis—Trough model with *diffusion-controlled glide* of dislocation core: Section 5.3 in [6]—assumes a stress-induced shortening of L [64]. Both effects decrease V with an increase in stress, resulting in a higher SRS (Eq. (2)) even without any changes in obstacle type or profile. In our prior modeling framework [6], ΔG_H scales almost linearly with the concentration of locally segregated H along the dislocation core. Hence, a higher H concentration necessitates a higher applied stress to overcome the obstacle, shifting the activated path to upside of the potential hill (i.e., smaller d , Fig. 8 (a-2) versus (b-2)). Consequently, both SRS and the *Haasen plot* intercept increase with H concentration—underlying the characteristic change (I) in Section 3.4, observed when relaxation was measured near the flow stress level (Regimes 1 in Fig. 4).

Fig. 8 (a-3) and (b-3) offer the same context from an alternative perspective, illustrating how the situation changes upon crosshead-holding, and how available ΔG stochastically increases with the aid of time (more rigorously, how a longer time available for deformation increases the probability that a dislocation can overcome an obstacle even when a larger activation barrier, ΔG , remains). Under high stress and correspondingly small d , a modest increase in ΔG (area shaded with dark gray in Fig. 8 (a-3)) prompts a sharp stress drop (i.e., greater and faster stress relaxation [6]), while the necessity to overcome larger ΔG to sustain deformation under a reduced stress level progressively renders the relaxation strain rate slower (cf. Eq. (7)). This stress drop induces pronounced increases in d and V (Fig. 8 (a-3)), and a corresponding decrease in SRS (cf. Eq. (2)). Such behavior was evident at high H concentration, where significant stress-dependent reduction in the *Haasen plot* intercept was observed (Fig. 5(b)). In contrast, at low H

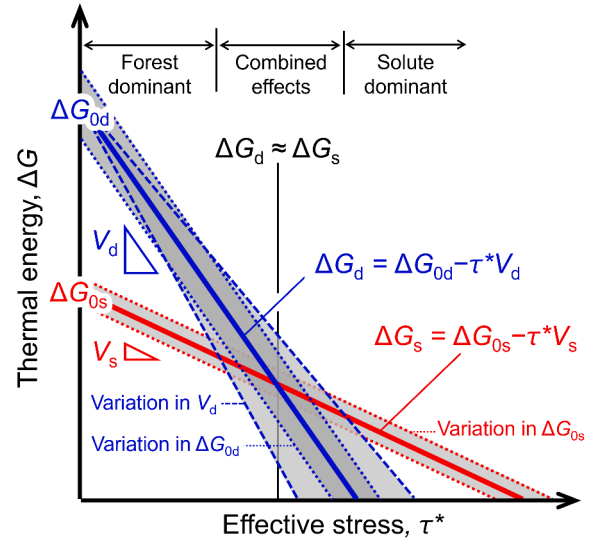


Fig. 9. Stress-dependent change in rate-controlling obstacles in materials containing solutes and forest dislocations, reproducing the schematic described by Schoeck [31]. A straight line for each obstacle type can be drawn if the obstacle has a square-shaped force-distance profile, while the line should become downward-convex asymptotes for any realistic obstacle profiles with finite slope like Fig. 8.

concentration, the initial stress already lies in a downside of obstacle potential with originally large d and V (Fig. 8 (b-3)). Thus, despite similar time-aided increase in ΔG , the resulting incremental fraction in d and V are minor, leading to only a slight reduction in the *Haasen plot* intercept during relaxation (Fig. 5(b)).

Given the condition of $\tau = \tau_y$, Eq. (3) yields $kT/V = m_y \tau_y$. Thus, plotting the yield stresses, τ_y , on the *Haasen plot* lines under three H concentrations provides the precise value of m_y by its slope. Taking a linear regression of the three cross marks in Fig. 5(b), m_y is substantially larger than the slope of whole the *Haasen plot*, m_d , inferring the

difference between solute atoms and forest dislocations in their character as short-range obstacles to dislocation motion. The classical formulation of thermally activated plasticity [65] offers the following power law dependence of the flow stress on strain rate:

$$\tau/\hat{\tau} = (\dot{\gamma}/\dot{\gamma}_0)^m \quad (8)$$

Here, $\hat{\tau}$ is the flow stress under the absence of thermal activation effect (i.e., top of the obstacle profile in Fig. 8). Combining with Eq. (7), one can obtain:

$$m = -\frac{kT}{\Delta G} \ln(\tau/\hat{\tau}) \quad (9)$$

Kocks, Argon, and Ashby formulated the universal form of activation energy as [22]:

$$\Delta G = \Delta G_0 \{1 - (\tau/\hat{\tau})^p\}^q \quad (10)$$

From eqs. (8)–(10), the relative rate sensitivity, m , is finally expressed as follows:

$$m = -\frac{kT}{\Delta G} \ln \left\{ 1 - (\Delta G/\Delta G_0)^{1/q} \right\}^{1/p} \quad (11)$$

Since the exponents p and q are in the ranges of 0~1 and 1~2, respectively [22], Eq. (11) necessarily derives smaller m for the obstacles having larger zero-stress activation energy, ΔG_0 , as for forest dislocations in the present case. The relationship $m_y \gg m_d$ substantiates the premise made in Section 1 that solute atoms are weak in their obstacle nature, while forest dislocations act as relatively strong barriers.

4.2.2. Decrease in slope

The observed reduction in *Haasen plot* slope apparently suggests a decrease in m_d , the parameter representing the work required for mobile dislocations to cut through forest dislocations [21,23]. This interpretation was implemented in our previous work [8]; however, the stress-dependent slope transition shown in Fig. 5(b) rules out such a permanent change in m_d . Rather, as stress relaxes, both non- and H-charged specimens converged into nearly identical slopes, indicating that this terminal slope reflects m_d truly governed by dislocation-dislocation interactions. A solute-induced reduction in *Haasen plot* slope has also been reported and discussed by Curtin [25]. He proposed that solute atoms, being more rate-sensitive than forest dislocations, could predominate not only the *Haasen plot* intercept but also its slope. Although his model predicts an inverse relationship between slope and stress (particularly the effective stress component associated with solutes), it does not explicitly incorporate the stress-dependence of the *Haasen plot* intercept, and thus it cannot account for the simultaneous decrease in intercept and increase in slope observed in Fig. 5(b). Alternative models have been offered, including those by Arsenaault and Cadman [19], Schoeck [31], Picu et al. [18], and Dong and Curtin [17], which consider the averaged or competitive effects of obstacle types differing in strength and density. Among these, Schoeck's simple model [31] most straightforwardly accounts for the experimental trend in Fig. 5(b).

Fig. 9 reproduces the description by Schoeck [31], where subscripts s and d refer to solute- and forest dislocation-related parameters, respectively. In general, the larger ΔG_0 and wider interspacing of forest dislocations lead to a greater activation volume, V , compared to solutes, allowing Eq. (6) to draw two mutually intersecting straight lines for their stress-dependent ΔG . For realistic obstacle potentials with finite slope, these lines bend into downward-convex asymptotes [17,22] since V is also stress-dependent, although the qualitative trend remains unchanged. On slip planes populated by multiple obstacle types, the glide kinetics is dictated by the obstacle having larger ΔG [16,18,31]. Thus, the most salient feature in Fig. 9 is the counterchange in dominance of solutes, ΔG_s , and forest dislocations, ΔG_d , at the crossover point between the red and blue solid lines. The crossover between ΔG_s and ΔG_d occurs only when the intercept on the horizontal axis of ΔG_s is larger than that

of ΔG_d (i.e., $\Delta G_{0s}/V_s > \Delta G_{0d}/V_d$) [31]. Nonetheless, such a situation is likely to be realized especially for the H-charged condition since the addition of the extra activation barrier, ΔG_H (Section 4.2.1), potentially shifts the overall ΔG_s line in an upward direction.

Above the crossover stress (i.e., the intersection between red and blue lines) in Fig. 9, ΔG is governed exclusively by solutes, ΔG_s . Moreover, since the density of solutes is overwhelmingly higher than forest dislocations throughout the deformation, ΔG_d no longer contributes significantly to glide kinetics at such sufficiently high stress level. The dominance of solutes may obscure the influence of forest dislocations, making the activation parameters, V and SRS, insensitive to strain-hardening (i.e., dislocation density). In H-charged Type310S steel, this condition may result from an additional effective stress induced by H (Fig. 1(a)). Consequently, as the stress level increases during V and SRS measurements under a given dislocation structure, the forest dislocation effect—reflected in the *Haasen plot* slope—is progressively reduced (Fig. 5(b)). In practice, both ΔG_d and V_d are statistically distributed due to the varied characters and spacing of intersecting dislocations, besides ΔG_s also have finite variation owing to the fluctuated solute distribution. With increasing stress, segments pinned by forests with higher ΔG_d and smaller V_d are sequentially activated, although experiments detect only their average response at each stress. The lines of ΔG_s and ΔG_d actually span a finite range, as indicated by the shaded area in Fig. 9. Thus, the shift from ΔG_d to ΔG_s dominance occurs gradually, not abruptly, across the “combined effects” regime at the center of Fig. 9. By contrast, as the stress decreases, ΔG_d eventually becomes larger than ΔG_s , making forest dislocations the more rate-controlling obstacles. Nevertheless, during motion between forest dislocations, a dislocation inevitably encounters numerous solute atoms and is therefore expected to experience resistance from both solutes and forest dislocations. This situation may correspond to the commonly observed *Haasen plot*, which exhibits a positive intercept and a significant slope.

In Regimes 2 and 3 (Figs. 4 and 5), where the elapsed time after the onset of relaxation was relatively long, factors such as dynamic recovery and strain aging may also affect the measured activation parameters. Nevertheless, when dynamic recovery influences the shape of the *Haasen plot*, it generally causes an upward deviation from the linear trend [30], whereas all the present plots in Regimes 2 and 3 clearly retain their linearity. Furthermore, as shown in Fig. 6(b), the slope of *Haasen plot* evolves primarily with stress rather than with relaxation time. This indicates that, although the recovery cannot be disregarded strictly, it is at least not significant enough to explain the slope change observed in the present study. Even for strain aging, its contribution was already shown to be negligible in our previous paper [6] through static aging tests and related analyses.

4.3. Stress-dependence of rate-controlling obstacles

4.3.1. Solute versus forest dislocations

The transition from ΔG_s - to ΔG_d -dominated behavior is illustrated more graphically in Fig. 10, depicting the interplays between solutes and forest dislocations on the slip plane. The illustration adapts the classical concept proposed by Kocks, Argon, and Ashby [22], which has since been extended in modeling efforts by the present authors [6,7] and others [17,18]. Briefly, a mobile dislocation segment, anchored at both ends by junctions with forest dislocations, bows out in adapting to applied stress with the angle, φ (Fig. 10(a)), until thermally activated collapse of junctions takes place. Throughout this bowing process, a field of dense solutes (i.e., weak obstacles) continuously resists the advancing segment, while the self-stress of curved dislocation imposes a line tension force, $Gb^2 \sin \varphi$, on the junctions (Fig. 10(a)) [22]. This line tension force aids in breaking the junctions (i.e., gradually reduces ΔG_d through the bow out process), enabling long-range motion of the whole dislocation segment *via* cutting through the forests when ΔG_d decreases to the level that thermal activation becomes a realm of possibility within a realistic time window.

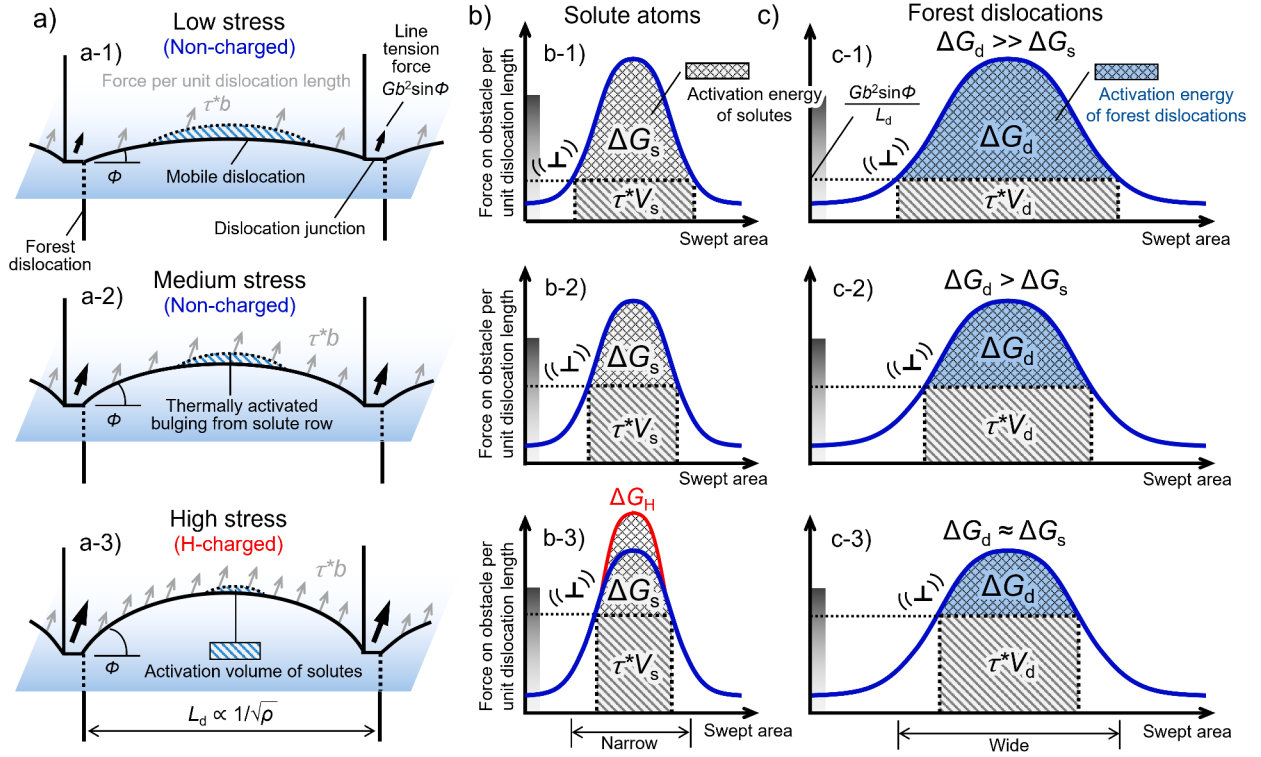


Fig. 10. (a) Schematic illustrations of the dislocation configurations under different levels of applied stress. The stress-assisted bow out of mobile dislocation segment between the anchoring points of the junctions with forest dislocations effectively reduces the corresponding energy barrier, accordingly making the collapse of junctions easier. (b)(c) stress-dependent changes in activation energy for (b) solutes and (c) forest dislocations.

The schematic profiles in Fig. 10(b) and (c), combined with Fig. 10(a), illustrate the stress-dependent dislocation position along the energy barriers posed by solutes and forest dislocations. Here, larger ΔG_0 of forest dislocations (Section 4.2.1) is expressed by its broader profile, though the peak heights of the profile remain ambiguous. The area—instead of distance—swept by dislocation during the activation event is used for the horizontal axis for cancelling the difference in segment length involved in overcoming each obstacle type. When the mobile dislocation encounters forest dislocations under low stress where $\Delta G_d \gg \Delta G_s$ (Fig. 10 (b-1) and (c-1)), the probability of the dislocation to overcome forest junctions is much smaller than that for solute barriers owing to the exponential dependence of plastic strain rate (i.e., dislocation velocity) on ΔG in Eq. (7). As a result, the dislocation remains to slightly bow out between the forest anchoring points after surmounting the solute field (Fig. 10 (a-1)) until relatively large ΔG_d is stochastically supplied by thermal fluctuation. The waiting time required to break the forest junctions is thus significantly longer than that to bypass individual solutes, making the glide kinetics mainly governed by ΔG_d . With increasing applied stress (Fig. 10 (b-2) and (c-2)), the upward shift of dislocation position along the obstacle profile reduces both ΔG_d and ΔG_s , with ΔG_d diminishing more rapidly due to the broader energy profile of forest junctions. This effectively reduces the incubation time required for the junction collapse and weakens the predominance of ΔG_d by narrowing the gap between overcoming probabilities of solutes and forests. A more extreme situation is met when the activation process involves H-induced extra energy barrier, ΔG_H , in ΔG_s that further elevates the stresses acting on the dislocation segment encountering these two types of obstacles (Fig. 10 (a-3), (b-3), and (c-3)). That is, the energetical relationship eventually becomes $\Delta G_s \approx \Delta G_d$, enabling the overcoming of forest junctions with a probability identical to that of solutes. Such a specific situation, where ΔG of solutes and forest dislocations become mutually equal (i.e., the crossover stress in Fig. 9), has been designated as the “critical condition” in [20,22]. As the stress

increases further, it is possible that the magnitude relationship between ΔG_s and ΔG_d is even reversed. Under these circumstances, the mobile dislocation barely feels the glide resistance by forest junctions that might be masked by solutes background. Ultimately, ΔG_s -dominated behavior is reached at high effective stress with a decreased slope of the Haasen plot, m_d . Note that the situation in Fig. 10 (a-3), (b-3), and (c-3) is not limited to the H-charged specimen, but rather probable also in the non-charged case if much faster strain rate that enables an ideally high effective stress level for $\Delta G_s \approx \Delta G_d$ is employed. The role of H is to realize such critical condition, which was inaccessible in the non-charged case, even under slow strain rate owing to the introduction of ΔG_H term.

The decrease in m_d seems straightforward as well if one refers to Eq. (9) again. Given that $\hat{\tau}$ in Eq. (9) is now the stress to bypass forest dislocations, which is not affected by H, without thermal activation effect, an increase in overall stress, τ , by solutes inherently leads to a reduced m value when comparison is done under an identical strain rate with fixed ΔG (i.e., Regime 1 in Fig. 5(b)).

4.3.2. Upper limit of activation volume

To describe the motion of local dislocation segments between anchored ends, we previously adopted the Trough model of SSH [64]. Originally proposed by Kocks, the model addresses SSH in concentrated solid solutions where neither the point-like obstacle approximation [62, 66] nor Labusch-type interactions [67] are applicable. A dislocation segment is stabilized by collective interactions with solutes within its cylindrical interaction volume, effectively forming a solute “row” that pins the dislocation [64]. From such energy minima—or trough, the dislocation advances via thermally activated excursion of a small bulge (Fig. 10 (a)), akin to breaking away from Cottrell atmosphere [66] or overcoming Peierls valley via kink-pair mechanisms [24]. The material volume involved in this bulging event determines the corresponding solute-related component of activation volume (Fig. 10(a-3)), and in

turn, strain rate sensitivity, $m_y\tau_y$. When H atoms segregate along the dislocation line, the activation energy for bulge nucleation increases by ΔG_H , requiring higher stress to sustain the dislocation's mobility [6,7].

Similar to classical models of atmosphere breakaway and kink-pair motion, the critical bulge configuration required for an advancement of whole dislocation segment is stress-dependent. As stress decreases, the bulge lengthens while its forward excursion also increases [22,64], as illustrated in Fig. 10 (a). Eventually, the bulge spans the distance between the forest anchoring points, constraining V by the spacing of forest dislocations [64]. In this regime, detectable activation parameters are governed entirely by the spacing of forest junctions, effectively masking solute effects in reversal. This forest-controlled behavior corresponds to Regimes 2–3 in the non-charged specimen, where the *Haasen plot* intercept approaches close to the origin (Fig. 5(b)). The near-zero intercept signifies the disappearance of solute effects at low stress, rendering the material behavior akin to that of pure FCC metals [21,23].

In the above context, it is instructive to quantitatively compare the experimentally determined activation volume, V (Fig. 5(a)), with the mean spacing of forest dislocations estimated from the total dislocation density, ρ . Empirically, this spacing is approximated as $\rho^{-1/2}$ in the forest hardening theory [30,68–70], forming the basis of the well-known Bailey–Hirsch relation [68]. Ito et al. recently investigated dislocation density evolution in Type310S steel with and without solute H using in-situ neutron diffraction during tensile deformation at room temperature [10]. They reported ρ of $\approx 10^{14}/\text{m}^2$ ($\rho^{-1/2} \approx 100$ nm) and $\approx 10^{15}/\text{m}^2$ ($\rho^{-1/2} \approx 30$ nm) at true strains of 0.10 and 0.20, respectively, with negligible influence of 7600 at ppm H. Assuming the Burgers vector $b \approx 0.25$ nm in Fe–Cr–Ni austenitic steels and sufficiently limited forward excursion of the anchored dislocation during its activation event, these values estimate $\approx 400b^3$ and $\approx 130b^3$. In the authors' previous study, dislocations in Type310S steel readily exhibited diffusely developed cell structures at a strain of 0.10 at 296 K [7]. Those cells evolved into more organized form at a 0.30 strain as confirmed in Fig. 7. Given that dislocation bowing (Fig. 10(a)) likely takes place in cell walls [57], the actual length of bowing segment may be somewhat smaller than $\rho^{-1/2}$. Indeed, the theory developed by Kuhlmann-Wilsdorf postulated the link length of dislocations bearing the deformation in cell structure as $L_d \approx \rho^{-1/2}/3$ [69]. Thus, $V \approx 250b^3$ at a strain of 0.10 in the Regime 3 of non-charged specimen (Fig. 5(a)) is in fair agreement with the estimation. Notably, the estimation is more accurate for a strain of 0.20, where V asymptotically reached around $130b^3$ (Fig. 5(a)), demonstrating the notion in original trough model by Kocks that the extent of V is finally confined by forest dislocation spacing [64].

4.4. Apparent similarity with other solute elements

An increase in *Haasen plot* intercept and a decrease in slope (Fig. 5 (b)) are not unique to H but are commonly observed in various FCC metals and alloys upon the addition and increasing concentration of solid solution-hardening elements [21,25,28]. The increase in intercept is typically attributed to the involvement of additional, rate-sensitive obstacles [21,25]. However, H atoms notably act in a different way from those conventional solutes; they primarily increase the activation energy for dislocations to surmount pre-existing alloying elements [6]. The reduced slope, on the other hand, has been linked to an increased work required for a mobile dislocation to cut through forest dislocations [21] or strain aging due to solute diffusion [71]. For example, certain solute species lower the stacking fault energy (SFE), widening the stacking fault ribbon and demanding extra work to constrict extended dislocations before intersecting with forests [21,38,72]. While H has been believed to reduce SFE in Fe–Cr–Ni austenitic steels [73–76], our *in-situ* neutron diffraction study on Type310S steel with ~ 7600 at ppm H showed no change in stacking fault probability across $\{111\}$ planes [10]. Along with the negligible impact of H on dislocation density (Section 4.3), this suggests that the average width of extended dislocations

remains unaffected. The absence of any H effect on dislocation–dislocation interaction strength was also demonstrated in Ref [10], where the amount of strain-hardening at a given dislocation density was the same in the non-charged and H-charged specimens (i.e., the dislocation-strengthening factor in the Bailey–Hirsch equation remained unchanged).

Solute diffusion and interactions with deformation-induced vacancies may also lower the *Haasen plot* slope [71,77,78], potentially due to dynamic dislocation pinning that introduces negative strain rate sensitivity. Some experiments and atomistic simulations indeed envisaged the stability of H-vacancy complexes and their function of resisting dislocation movement [79,80]. Such diffusion-related effect would cause the slope to decrease with relaxation time. However, the trend in Fig. 5(b) showed the opposite behavior, thereby reinforcing the interpretation developed in Sections 4.2–4.3 by excluding these secondary contributions.

5. Conclusions

Thermally activated deformation of Type310S (Fe–24Cr–19Ni) austenitic steel containing ~ 8500 at ppm H was investigated using stress relaxation tests with durations up to 1000 s. The main focus was on the competition between solutes, including both alloying elements and H, and forest dislocations in determining deformation kinetics and activation parameters. A regime-wise analysis was introduced, which involved extracting selected stress levels from relaxation curves to assess the stress-dependent variations in *Haasen plot* (flow stress vs. strain rate sensitivity). Back stress evolution was also evaluated through unloading after relaxation, leading to the following conclusions:

1. Under a given strain rate, H-charged specimens consistently exhibited higher flow stresses than non-charged specimens, resulting in an almost parallel upward shift of the stress-strain curves. The elevated stress in H-charged specimens can primarily be attributed to the increased effective stress caused by H, with minimal effects from back stress, except in the large strain where deformation twinning intervened.
2. When relaxation behavior was assessed under H-induced elevated effective stress levels, two prominent changes were identified in the *Haasen plot* shape: (i) an increase in intercept and (ii) a decrease in slope. These two features were more pronounced with increasing H concentration, accurately reproducing our previous findings from short-term (~ 30 s) stress relaxation tests.
3. The intercept increase was found to reflect the function of H in making pre-existing alloying elements more rate-sensitive by introducing an additional energy barrier for dislocations to surmount these intrinsic obstacles. The slope decrease was attributed to the weakened influence of forest dislocations on glide kinetics, with solutes primarily controlling activation parameters at sufficiently high effective stress levels.
4. The H-induced changes (i) and (ii) were gradually diminished as the effective stress is relaxed. This suggests that the impact of H on *Haasen plot* is not permanent but is rather stress-dependent, with the effects on activation parameters being most pronounced under high-stress conditions. These findings highlight the competitive nature of dislocation-obstacle interactions, where H, alloying elements, and forest dislocations play roles in a stress-dependent manner to control deformation behavior.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

CRediT authorship contribution statement

Yuhei Ogawa: Writing – original draft, Methodology, Investigation, Funding acquisition, Conceptualization. **Akinobu Shibata:** Writing – review & editing, Supervision, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.actamat.2026.122577](https://doi.org/10.1016/j.actamat.2026.122577).

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