Quantifying the effects of tempering on individual phase properties of DP980 steel with nanoindentation

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Abstract

Tempering treatment is conducted on a commercial dual phase (DP) 980 steel at 250 °C and 400 °C for 60 minutes each. Ferrite and martensite grains are distinguished using electron backscatter diffraction (EBSD) and scanning probe microscopy (SPM), and the martensite volume fractions (MVF) are determined based on the image quality (IQ) map. Indentation tests combined with a newly developed inverse method are used to obtain the individual phase flow properties in each sample. The results show that, i) tempering significantly reduces martensite yield strength, while it slightly reduces the ferrite yield strength; ii) tempering temperature has a more significant influence on the work hardening exponent of ferrite than that of martensite. As a validation, a simple rule-of-mixtures is used to verify the above-predicted individual phase flow stresses with the experimentally obtained overall true stress vs. true strain curves.

Keywords: Nanoindentation; Plastic flow properties; Tempering; DP steels

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1. Introduction

Advanced high-strength steels (AHSS) are increasingly being used by the global automotive industry to simultaneously reduce vehicle weight and improve occupant safety [1-3]. Among different grades of AHSS, dual phase (DP) steels are the most popular commercialized products because their micro-constituents, i.e., ferrite and martensite (or lower bainite), work together to deliver high strengths and good ductility. DP steels are designated with their minimum ultimate tensile strength (UTS), and commercial DP steels have reached a UTS value of 980 MPa, thus named DP980 [4]. Various chemical compositions and thermo-mechanical processing strategies are typically used by different steel companies to produce DP980 steels that satisfy the minimum UTS criterion, and they typically lead to significant differences in the steels' microstructures [5].

Rapid cooling rates (quenching) employed in the thermo-mechanical processes by steel production facilities can leave certain DP steels with high strength disparity between the two constituent phases, leading to limited overall ductility and local formability such as hole expansion ratio. To improve formability, a tempering heat treatment process is often used to soften the microstructure by relieving some portion of the carbon in super-saturated martensitic phase so as to decrease the difference between mechanical properties of ferrite and martensite [6]. Tempering is known to reduce the flow stress and increase overall ductility of DP steels by delaying the deformation localization [7]. Since tempering is a diffusion-controlled process involving the re-arrangement of carbon atoms in the microstructure, both temperature and time are expected to influence the tempering response following the Hollomon-Jaffe relationship [6, 8]. Large ranges in temperature (from 100 °C to 500 °C) and tempering duration (from 20 s to 10^7 s) have been used to investigate the influence of heat treatments, and the results have shown that the tempering temperature plays a more dominant role [9-22].

In the literature, the effects of tempering on various macro-scale mechanical properties (i.e. UTS, elongation, hardness, and formability) [10, 14, 20] and microstructural-scale features (i.e. grain orientation, martensite morphology and grain size) [12, 15, 17, 21] have been reported. Overall reduced

flow-stress were observed for tempered steels [22, 23], and martensite softening [15, 19, 24] has also been studied. Various experimental techniques or apparatus such as X-ray diffraction (XRD), scanning electron microscopy (SEM) or transmission electron microscopy (TEM) have been used to understand the relation between the carbide movement in martensite and heat-treatment temperatures [25]. To date, most of the tempering studies report on the overall performance of the tempered materials and only a few address how individual phase (ferrite and martensite) properties are affected by tempering [9, 26]. Even though the reduction of strength disparity between two phases was believed to lead to better formability of DP steels, none of the previous studies cited have attempted at quantifying the effects of tempering on the changes of individual phase properties of a DP980.

To interrogate the individual phase flow properties of DP steels, fine grain nanoindentation [5, 27, 28], micro/nanopillar compression [29, 30], in-situ neutron diffraction [31] and high-energy X-ray diffraction (HEXRD) [32, 33] have all been reported as direct and indirect measurement techniques. Among them, nanoindentation can be considered as the most convenient method due to the factors including simple sample preparation, wide equipment availability, as well as the statistical nature of the data generated. For example, one recent study on a tempered DP980 has pointed to a decrease in martensite hardness after tempering [15]. However, the quantitative connections between nanoindentation responses, e.g. load-depth curves and hardness, and the individual phase properties of tempered DP steels were not established. Recently, nanoindentation together with an inverse method has been developed to extract the phase properties of a complex quenching & partitioning (Q&P) steel [27]. In the present study, we employ a similar indentation and inverse calculation technique aimed at quantifying the effects of tempering temperature on the individual phase flow stress of a commercial DP980 steel.

Heat treatments are conducted at two temperatures (250 °C and 400 °C) well below the eutectoid temperature (727 °C) so that the MVF and average grain size remain unchanged. A multi-scale indentation and inverse calculation technique [27] combined with a SPM and EBSD analysis [34] are used to calculate the flow stress of ferrite and martensite for each temper temperature. As a verification, the calculated individual phase flow behaviors are used to estimate the overall stress-strain curves of the

as-received and tempered samples with rule-of-mixtures, and the results are compared with experimentally obtained tensile curves with good agreement. The applicability of the proposed methodology and the evolution of individual phase property due to tempering treatment of DP980 samples are discussed. The methodology and the corresponding results shown in this study can help guide the selection of tempering parameters in optimizing the mechanical properties of DP steels for their intended applications.

2. Experiments

2.1 Sample preparation and heat treatment

In this study, the as-received material is a commercial low-carbon DP980 steel sheet with 1.18 mm thickness and a chemical composition as listed in Table 1. To avoid rolling-induced edge effect, all tensile samples were cut from the center of the sheet. Heat treatments were performed on the as-received DP980 samples at 250 °C and 400 °C for 60 minutes respectively followed by air-cool, see heat treatment schedule illustrated in Figure 1(a). Samples were mounted, grinded and polished with standard metallographic procedures, with DP980-AsR, DP980-250 and DP980-400 designating as-received, 250 °C tempered and 400 °C tempered samples, respectively.

2.2 Ferrite and martensite identification

In order to identify individual ferrite and martensite grains in a dual phase microstructure, SEM is typically used, and the differences in height after chemical etching are usually used in differentiating the martensite grains from the ferrite matrix [5, 35]. In this study, a recently developed method is utilized which utilizes EBSD IQ map and SPM in identifying the martensite and ferrite phases [36-38] with the observation that martensite phases in the dual phase structure usually have higher mis-orientation angle and geometry necessary dislocations (GNDs) density [36].

All metallurgical samples were mounted in bakelite and polished with standard techniques to a 0.25 μm diamond finish in which a 0.05 μm colloidal silica vibratory polishing was applied for 60

minutes. Once the vibration polishing step was completed, data pertaining to the microstructure was collected with a field emission SEM equipped with an EDAX (energy dispersive spectroscopy) EBSD detector. Measurements were performed at 20 keV beam energy with a 100 nm step size. Figure 2 shows the EBSD IQ maps of the as-received and the tempered samples, where the light grey regions represent ferrite grains and the dark grey regions represent martensite grains.

2.3 Multi-scale indentation tests

Multi-scale indentation tests were performed on each sample with four steps:

(1) Nanoindentation under SPM

Samples were polished with a PH 9-11.5 colloidal silica suspension in a vibratory polish. Since martensite grains etch faster than ferrite [36], different surface heights were detected in the SPM image, see the surface images in Figure 3 where the variations of surface heights are represented by a color gradient. In principle, the grains with the same phase and orientation should have the same heights in SPM images with the same polishing technique.

Nanoindentation with a small depth (in the order of ~ 50 nm) have been used as a quantitative technique in characterizing DP980 steels [5] with fine grains. In this study, nanoindentation tests were performed using a Hysitron TI 950 Triboindenter with a sharp Berkovich indenter tip operated in displacement-control mode. A constant indentation depth of 50 nm was used and the rate of loading was controlled to be 10 nm/s for both loading and unloading. A minimum of ten indents were intentionally placed on different grains of a single phase to include possible statistical distribution of the hardness values. The indents were located at the center of the grains to avoid boundary effects. The average hardness values for each phase obtained by indentation under SPM, as shown in Figure 3, are used to guide the subsequent data processing of the indentation load-depth curves obtained from the nanoindentation batch runs with 15x15 indent arrays.

(2) Nanoindentation batch run with 15x15 indent array:

To capture the statistical variations of the individual phase stress-strain response, a 15 x 15 nanoindentation array (i.e. 225 indents in total) was performed on each sample and the load-depth curves for all the indents were recorded. To be consistent with the nanoindentation tests under SPM, the indentation depth was programmed at 50 nm with the loading rate of 10 nm/s for both loading and unloading. A 5 μm indentation spacing was chosen to eliminate potential result interference by overlapping plastic zones.

(3) Indentation at various depths:

To quantify the well-known indentation size effect (ISE) of the DP steel samples, a series of 5x5 indentation array was performed on each sample. Under the displacement-control mode, indents were performed to 100 nm, 150 nm, 250 nm, 300 nm and 400 nm in depth, respectively. The indentation spacing was correspondingly increased with increasing indentation depth to avoid plastic zone overlap.

(4) Vickers hardness:

To complete the ISE and relate nanohardness and micro hardness of each sample, Vickers hardness data were obtained with a 300 grams mass and a 10s dwelling time on all samples. Ten Vickers indentations were randomly placed on each sample and the averaged hardness values were calculated and reported as Vickers hardness.

2.4 Tensile test

Dog-bone shape tensile samples were machined along rolling direction according to the ASTM standard (ASTM E8-04) with a $25.4 \text{ mm} \pm 0.762$ gage length and a $6.35 \text{ mm} \pm 0.127$ width. In the present study, the strain rate was controlled at 10^{-3} /s. An Instron tensile testing frame was used to perform tensile tests at room temperature, and three replicates in each group were performed under the same testing condition. The samples were gripped in order to ensure that the samples were aligned axially and to minimize bending. During the tests, the gage length elongation was recorded with a contact extensometer traveling with the samples, and all tests ended upon fracture. Figure 1(b) shows the load-elongation and

true stress-strain curves obtained. The elastic modulus was determined by fitting a linear function to the initial portion of the experimental data up to 0.2% strain. Values for the yield strength and UTS of each sample have been measured before [34], and the results are plotted in Figure 1(b). The plastic stress was fitted according to Hollomon equation $\sigma = K\varepsilon^n$, where σ is the true stress, ε is the true strain, n is the work hardening exponent, and K is the strength coefficient. The values of hardening exponents are summarized in Table 2.

3. Inverse calculation

To calculate the flow stress from nanoindentation load-depth curves, a three-step method proposed by Cheng et al. [27] was employed. Figure 4 illustrates the flowchart of the analysis process. The first step is to assume a power-law flow behavior and calculate the elastic modulus with the Oliver-Pharr method [39]. The second step is to determine the individual phase yield strength (i.e., martensite and ferrite) following the empirical relationship between yield strength and nanohardness (at 50 nm indentation depth) reported by Rodriguez and Gutierrez [40]. Finally, a representative stress, $\sigma_{0.033}$, is calculated from Dao's method that considers ISE and indenter bluntness effect in order to estimate the work hardening exponent [41]. More details on this method as well as its application on a multi-phase Q&P steel can be found in Cheng et al. [27].

3.1 Determining constants for the inverse calculation

The following four constants need to be identified for the inverse calculation:

- 1) Constants for contact area function: due to the extremely small indentation depth utilized in this study, i.e., 50 nm, strong indenter bluntness effects are anticipated, and hence must be taken into account in the calculation of contact area. The contact area (A_c) was determined from the effective indentation depth (h_c) for each sample with the method outlined in [39] to fit the polynomial Eq. (1) with fitting constants θ_i . The area function was calibrated before testing each sample, and the fitted θ_i are listed in Table 3.

$$A_c = \theta_0 h_c^2 + \theta_1 h_c + \theta_2 h_c^{\frac{1}{2}} + \theta_3 h_c^{\frac{1}{4}} + \theta_4 h_c^{\frac{1}{8}} + \theta_5 h_c^{\frac{1}{16}} \quad (1)$$

- 2) Constants for the slope of yield strength-nanohardness relationship: Rodríguez and Gutierrez [40] reported a linear relationship between the yield strength (σ_y) and hardness (H) of single phase martensitic and ferritic steels by performing nanoindentation tests with indentation depth at 50 nm:

$$H = a\sigma_y + b \quad (2)$$

Since ferrite and martensite are the two constituent phases in the current DP980 steel, and the current indentation depth is also set at 50 nm, the same constants a and b from Rodríguez and Gutierrez [40] are used in this study: $a = 5.2$ and $b = 1.3$ GPa.

- 3) Characteristic length (h^*) of ISE: the hardness value for each indent was calculated by the Oliver-Pharr method [39]. The hardness vs. indentation depth relationship for all the indents (i.e., ISE curves) of each sample were plotted (see Figure 5), and the characteristic length for the entire material was calculated with the fitting model proposed by Nix-Gao [42]:

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}} \quad (3)$$

where h is the indentation depth, H is the hardness at the corresponding depth, and H_0 is the hardness value outside the ISE regime as measured by Vickers hardness, see Figure 5. The characteristic lengths h^* for each sample are obtained by numerically fitting Eq. (3), and the corresponding values are summarized in Table 3. Note that characteristic length scale here is obtained by the overall response of each sample rather than the individual phase response [28] due to the small grains in the current study.

- 4) Loading curvature correction constant α (defined as $\frac{\bar{c}}{c_n}$ in Figure 4): in order to calculate the representative stress with Dao's method [41], a curvature correction factor α was

introduced by Cheng et al. [27] to correct the nanoindentation loading curvature considering ISE and indentation bluntness effect in order to calculate the representative stress ($\sigma_{0.033}$). Following the same procedure, the values of the correction constant α are determined and summarized in Table 3.

Once all the above parameters have been determined, the inverse method can be readily used to calculate the flow stress and the hardening exponent in the power-law relationship.

3.2 Correlating indents to phases

Before the above inverse calculations can be applied to the load-depth curves from the 15x15 indentation arrays obtained in a batch mode, analyses must be performed to identify the phase of each indent. Taylor et al. [5, 6] and Cheng et al. [27] utilized a direct visual-based method in correlating the indents to their individual phase identities under SEM. However, such an effort can be time consuming and infeasible for the large number of indents covered in this study. Similar to the indentation under SPM results shown in Figure 3, Taylor et al. [5] also reported that the lower bound of martensite hardness is much higher than the upper bound of ferrite hardness for steel D in their study, which is identical to the DP980-AsR sample in the current work. Taking advantage of no overlap between the hardness ranges of the two phases, it is possible to separate the martensite and ferrite phases in the hardness histogram (see Figure 6) utilizing the average hardness values obtained from indentation under SPM.

Due to the batch run nature of the 15x15 indentation arrays, some indents will inadvertently land on grain boundaries. In previous nanoindentation studies [27, 30], higher hardness values were observed near the grain boundaries and interphase regions. Considering the possible grain boundary and GND strengthening effects [27, 30], the range of ferrite hardness is determined from the lowest value in the hardness histogram (Figure 6) until the average hardness value equals the average hardness obtained with SPM. For example, if the average hardness of ferrite in DP980-AsR was found to be 4.78 GPa under SPM, all the indents from the 15x15 array having 4-5.5 GPa are considered as ferrite to obtain the 4.78 GPa

average hardness. Since the martensite grains are much harder than ferrite, the hardness range for martensite should start from the highest value in the hardness histogram until the average value matches the average hardness obtained under SPM. The same inverse calculation method was adopted for DP980-250 and DP980-400, and the identified ferrite and martensite indents are also illustrated in Figure 6.

4. Results

4.1 Characteristic length and correction factor

The characteristic length is numerically fitted with Eq. (3) and calculated to be 210 nm for DP980-AsR, 200 nm for DP980-250 and 160 nm for DP980-400. These values were adopted in the inverse calculations for each sample. The correction factor α calculated is 0.97 for DP980-AsR, 0.92 for DP980-250, and 0.84 for DP980-400. See results in Table 3. These values are smaller than that previously obtained for a multiphase Q&P steel with a more complex microstructure and smaller grains ($\alpha=1.35$), which indicates that the correction factor is a variable that depends on ISE and indenter bluntness.

4.2 Hardness histogram

The hardness histograms resulting from indentation array are shown in Figure 6 together with the hardness ranges of martensite and ferrite obtained with averaged phase hardness from SPM indentation results. It shows that the hardness distributions of ferrite are much narrower than those of martensite for all the samples. While ferrite grains have more uniform crystal structures [5], the large hardness variations of martensite in each sample may be attributed to its heterogeneous structures, variable carbon content in martensite and the possibility of carbides in martensite [7, 17, 19, 43].

Tempering induced overall hardness reductions are also illustrated with the shift of indent percentage for each sample as summarized in Figure 6. The martensite nanohardness values consistently shifted lower from the as-received material to tempered samples with increasing temper temperature. For example, no indent with hardness over 9 GPa is observed in the tempered samples. Much smaller hardness reductions are observed for the ferrite phases in all three samples.

4.3 Inverse results

Grain level heterogeneities include the variations in grain size, elemental distribution, grain orientation as well as influences of grain boundaries (ferrite-ferrite or ferrite-martensite) and GND, hence the mechanical behaviors of the material points under different indents are expected to be different [27, 30] for a given phase in each sample. As shown in Figure 6, at least 20 indents were used for each phase in all three samples in the inverse calculations with the iso-strain-based rule of mixture. Figure 7 shows the average flow curves of all the indents within each phase for all the three samples: the red solid curves and black solid curves denote the average flow curve of martensite and ferrite, respectively. Note that the weight of each indent on each phase is assumed to be 1, so the mean value of the phase property is the same as its weighted average. The average properties obtained for each phase including elastic modulus (E), yield strength (σ_y) and work hardening exponent (n) are listed in Figure 7 for all the samples.

5. Discussions

5.1 Tempering effects on individual phase properties

Since tempering is a diffusion process, different tempering temperatures will lead to changes in carbon and other alloy element (such as Si and Mn) [6, 7] distributions, as well as changes of dislocation densities in different phases. The overall changes of mechanical properties from tempering treatment can be explained in terms of the changes of individual phase properties obtained in the current study.

1). Significant softening of martensite

A significant flow stress reduction was observed for martensite of both tempering temperatures as shown in Figure 7: the yield strength of martensite was reduced by 286 MPa (~19%) for 250 °C tempering and 320 MPa (~22%) for 400 °C tempering. In the literature, significant differences have been reported on the internal structures of martensite grains between tempered and as-received DP steels [7, 17, 19, 43, 44]. The precipitation of ϵ -iron carbides was observed in the microstructures of martensite, and residual stresses and dislocation density were found to decrease after tempering at 250 °C [17, 23, 44]. Thus, part

of the solid-soluted carbon in martensite grains was removed which leads to flow stress decrease in martensite. When tempering temperature increased to 400 °C, the progression of the tempering effects on the microstructure at 250 °C will continue [44], yet the possible dislocation pinning by the precipitated carbides would prevent further martensite softening [44]. Thus, only a small amount of further martensite yield strength reduction, ~34 MPa, is observed in DP980-400 sample. Meanwhile, the n of martensite was almost unchanged, ~ 0.08 for as-received sample and tempered samples. Thus, similar martensite phase properties were obtained in the current DP steel after tempering at 250 °C and 400 °C, indicating that the 60 minutes tempering time used in this study is long enough such that the tempering temperature within 250-400 °C will only induce minor differences in the tempered martensite properties. Higher tempering temperatures, such as 600 °C, can further soften the flow stress of martensite until the martensite grains are fully decomposed [22].

2). Minor softening of ferrite

Different from martensite, ferrite grains have less carbon content than martensite and the softening in ferrite is relative minor [22, 44]: the ferrite yield strength was reduced by 41 MPa (~ 6%) and 103 MPa (~ 15%) for tempering temperatures of 250 °C and 400 °C, respectively. In ferrite, the tempering treatment at 250 °C can relax the residual stress and reduce the dislocation density [44] so that the flow stress of ferrite was reduced. When the tempering temperature increased to 400 °C, the further softening in ferrite could be attributed to the precipitation of carbides which lowers the solute carbon concentration in ferrite [45]. Besides yield strength, results in Figure 7 indicate that tempering treatment influences the work hardening exponent n of ferrite: the n value of ferrite reduced from 0.11 in DP980-AsR to 0.08 in DP980-250, and then increased to 0.13 in DP980-400. Although the n of ferrite has changed depending on the tempering temperatures, it is consistently higher than the corresponding martensite in both the as-received sample and the tempered samples.

3). *Tempering effect on elastic modulus*

The inversely calculated elastic modulus of martensite is slightly higher (6 GPa, ~3%) than that of ferrite in the as-received sample. This observation is reasonable since martensite typically contains more carbon than ferrite does, and the higher impurity concentration in martensite can lead to the increase of elastic modulus [46]. After tempering, the elastic modulus of martensite was 13% and 7% higher than that of ferrite when treated at 250 °C and 400 °C, respectively. Generally, various degrees of carbide precipitation have been reported when the tempering temperature was greater than 200 °C [7, 47]. Although carbon diffusion from martensite could potentially reduce elastic modulus during tempering, the newly generated carbides, such as Fe₃C₂ with very high elastic modulus (~295 GPa according to the calculation using density functional theory [46, 48]), could lead to overall higher elastic modulus. Here, the inversely calculated elastic moduli of martensite are consistently higher than those of ferrite in both the as-received sample and the tempered samples.

Thus far, the tempering effects on individual phase property changes under different tempering temperatures have been quantitatively obtained. The specific property values, including σ_y , n and E , of each phase are listed in Figure 7, where significant softening of martensite, minor softening of ferrite and the differences between elastic modulus of each phase are summarized.

5.2 *Validation of inverse calculation results with experimental tensile curves*

In the AHSS literature, multiple studies have used a simple rule-of-mixtures to estimate the overall strength of the composite DP steels with reasonable accuracies [13, 49, 50]. In rule of mixtures, the overall flow stress σ_{all} at a given strain can be estimated from the flow stresses of the individual constituent phases based on an iso-strain assumption:

$$\sigma_{all}(\varepsilon) = \sigma_F(\varepsilon)f_F + \sigma_M(\varepsilon)f_M \quad (4)$$

where f is the volume fraction of each phase, and the subscripts F and M denote ferrite and martensite, respectively. Here, f_F and f_M are determined to be 75% and 25% for all the three samples using the

combined EBSD and SPM techniques based on the image quality map processed with an image processing software detailed by Zhang et al. [34], and they remain unchanged during deformation since martensite decomposition was not observed at the tempering temperatures studied [34]. Figure 8 shows the comparisons between the rule of mixtures calculated σ_{all} (blue curves) with inversely calculated flow stress curves for σ_F (black curves) and σ_M (red curves) as inputs and the experimentally measured true stress-strain curves (magenta curves) for all the samples. Results in Figure 8 indicate that good agreements have been obtained between the rule of mixtures estimated and the experimentally measured true stress-true strain curves for all the three samples. In particular, there is a close match between the predicted and measured hardening behaviors before UTS for all three samples. Overall, the similarities of the overall flow properties for each sample confirm that the nanoindentation-based inverse method can be used to quantify individual phase properties for DP980 samples after tempering heat treatment.

Note that the martensite and ferrite volume fractions for each sample were determined using an image processing technique with a specific threshold value, hence they are deterministic under that threshold. For the same steel, however, Taylor et al. [5] reported a small amount of retained austenite in the microstructure (<1%) with EBSD, and the MVF was reported as 28%. Therefore, MVF measurement uncertainty (~10%) will directly influence the results of rule of mixture for the overall stress vs. strain curves.

5.3 Effects of tempering on local formability and tensile ductility

Results in Figure 7 and 8 show that both tempering temperatures are effective in decreasing the strength disparities between the ferrite and martensite phases, therefore both tempered steels are expected to have better local formability (i.e., hole expansion ratio) than the as-received DP steel as reported in literature [6, 23]. Even though both ferrite and martensite are softened under the two tempering temperatures, tempering in this study does not lead to an increase in the overall ductility, similar to findings on certain tempered commercialized DP steels in the literature [6, 13, 22]. The total elongation of DP980-250 is ~7%, less than ~10% of DP980-AsR; and the total elongation of DP980-400 is ~10%,

similar to that of DP980-AsR. This is because the two tempering temperatures lead to different hardening exponent n for the ferrite and martensite phases as discussed before. Since the ductile failure of DP steels can be explained as the result of plastic instability induced by the microstructural-level inhomogeneities during plastic deformation between the hard and the soft phases [51], materials with higher constituents hardening coefficients can be expected to yield higher ductility according to the Considr criterion. Compared to DP980-AsR, tempering at 250 C kept the n value for martensite almost unchanged, but significantly reduced the n value for ferrite due to the possible precipitation of fine carbides ($\text{Fe}_{2.3}\text{C}$) during tempering [7, 17]. This leads to the noticeable reduction in overall ductility of DP980-250. Further increasing the tempering temperature to 400 C does not change the martensite hardening exponent n , but increases the ferrite n value to 0.13, thus restores the tempered ductility to 10%.

Even though yield strength-hardness and UTS-hardness correlations have been established by previous research [40, 52], the current work on DP980-250 and DP980-400 indicates that nanohardness alone may be insufficient in capturing the overall mechanical properties in characterizing tempered samples [15, 19, 24]. The full stress vs. strain curves of individual phases, in particular the plastic flow stress, are determined based on elastic modulus, yield strength as well as work hardening exponent [53]. Thus, it is crucial to utilize the load-depth curves from nanoindentation in conjunction with the inverse method to calculate work hardening exponent in order to obtain the comprehensive phase properties.

5.4 Correlation between n and ISE

A generally relationship between work hardening exponent n and natural logarithm value of h^* normalized by the magnitude of Burgers vector, $\ln(h^*/b)$, has been reported by Kim et al. [54] from testing 22 metallic materials including Ni and SCM21 steel by introducing pre-strains to change the ISE of as-received materials. As mentioned above, tempering reduces the total dislocation density [21], including GNDs density and statistically stored dislocations (SSDs) density, within the DP microstructures [36], leading to lower nanoindentation hardness for the tempered samples. With the numerical fitting with Nix-Gao model [42], the characteristic h^* was also observed to decrease with

higher tempering temperatures. Since ISE is generally supposed to be associated with GNDs [42, 55, 56], the n vs. $\ln(h^*/b)$ relationship for different tempered samples are plotted in Figure 9 together with the pre-strained SCM21 steels reported by Kim et al. [54] with the n and h^* values for each sample listed in Table 3. A similar trend can be observed for the tempered DP980 samples tested in the current study: the sample with higher $\ln(h^*/b)$ values also has a higher n value in general. The results hence indicate that the correlation between n and $\ln(h^*/b)$ as reported in [54] are valid for material processes including straining and heat treatment.

6. Conclusion

In the present study, nanoindentation tests, EBSD and SPM were adopted to analyze the tempering effects on the individual phase properties of a commercial DP980 steel. The ferrite and martensite grains were separated with EBSD and SPM, and the nanohardness of ferrite and martensite grain is obtained under SPM. The average phase properties for each sample are calculated with previously developed inverse method using experimental nanoindentation load-depth curves, and the inverse results were verified with the true stress- true strain curves from tensile tests.

It is demonstrated that the current inverse method can provide individual phase properties for each sample. Thus, a quantitative relationship between the tempering temperatures and individual phase properties in DP steel can be established:

- (1) Tempering at 250°C reduces martensite and ferrite yield strength by 286 MPa (~ 19%) and 41 MPa (~5%), respectively; while tempering at 400 °C reduces martensite and ferrite yield strength by 320 MPa (~22%) and 104 MPa (~18%), respectively;
- (2) Tempering does not influence the work hardening of martensite at either 250 °C or 400 °C; while tempering at 250 °C reduces the work hardening of ferrite from 0.11 to 0.08, and tempering at 400 °C increases the work hardening of ferrite to 0.13;

- (3) The average elastic modulus of martensite is higher than that of ferrite in both as-received sample and tempered samples.

It should be noted that the indent phase identification method used in this study would be not applicable to higher temperature heat-treated steels due to potential hardness range overlap of different phases. In addition, the accuracy of the inverse method depends significantly on the accuracy of the yield strength-hardness empirical relationship. With the current nanoindentation tests and inverse calculation, the individual phase properties of multi-phase steels after tempering can be accurately predicted. Since the mechanical performance of DP steels are determined by the individual phase properties, the current study provides a quantitative method in analyzing the effect of tempering treatments on DP steels, and it enables steel producers, fabricators, and end users to optimize their heat treatment parameters (i.e., time and temperature) to obtain the mechanical properties for the desired applications.

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Tables

Table 1 Chemical composition of DP980 in the present study [5]

Chemical composition	Al	C	Cr	Cu	Mn	Mo	Ni	P
wt %	0.05	0.12	0.25	0.01	2.47	0.36	0.01	0.014
Chemical composition	S	Si	Ti	B	N	Nb	V	Zn
wt %	0.04	0.03	0.01	0.01	0.009	0.02	0.01	0.01

Table 2 Parameters obtained from tensile test of each sample

Sample	DP980-AsR	DP980-250	DP980-400
E (GPa)	195	202	206
UTS (MPa)	1118 (6.8 %)	993 (5%)	984 (6.0%)
n	0.105	0.082	0.074

Table 3 Parameters used for nanoindentation inverse calculation of each sample

	θ_0	θ_1	θ_2	θ_3	θ_4	θ_5
Contact area function	24.5	0.12	0.25	0.01	2.47	0.36

Sample	DP980-AsR	DP980-250	DP980-400
$h^*(nm)$	210	200	160
α	0.97	0.92	0.84

Figures

Figure 1 (a) The tempering time and temperature for each sample and (b) The true stress- true strain curves from tensile test

Figure 2 The EBSD IQ maps of DP980-AsR, DP980-250 and DP980-400

Figure 3 The SPM image of each indentation and the corresponding hardness on each phase on DP980-AsR: F denotes indents on ferrite grains (bright regions), M denotes indents on martensite grains (dark regions) and FM denotes the indents on or close to grain boundary

Figure 4 Flowchart of the inverse method applied in the current study (more details on parameter definitions can be found in [27])

Figure 5 ISE of current samples (solid dot) and fitted curve (dotted curve) with Nix-Gao model [42]

Figure 6 Hardness histogram for nanoindentation on different samples: (a) DP980-AsR, (b) DP980-250 and (c) DP980-400

Phase		E (GPa)	σ_y (MPa)	n
AsR	F	211±11	682	0.11
	M	217±15	1510	0.08
250	F	212±13	641	0.08
	M	240±35	1224	0.07
400	F	217±30	579	0.13
	M	232±36	1190	0.08

Figure 7 Inverse calculated average phase properties and the corresponding properties values (F denotes Ferrite and M denotes Martensite in the properties table)

Figure 8 Comparison of rule-of-mixture estimated flow stresses with inverse results and experimental stress-strain curves: (a) DP980-AsR, (b) DP980-250 and (c) DP980-400

Figure 9 Relationship of n vs. $\ln(h^*/b)$ for DP980 (black square) in the current study and SCM21 (red dot) from Kim et al. [54] (SCM21_2 denotes SCM21 steel with 2% pre-strain and SCM21_5 denotes SCM21 steel with 5% pre-strain; $b=0.246\text{nm}$)

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