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

G. Cheng [a,1], F. Zhang [b,1], A. Ruimi [c], D.P. Field [b], X. Sun [a,*]

[a] Physical and Computational Sciences Directorate, Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA, USA
[b] School of Mechanical and Materials Engineering, Washington State University, Pullman, WA, USA
[c] Department of Mechanical Engineering, Texas A&M University, Doha, Qatar

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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 min 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.

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 supersaturated 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 rearrangement 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

* Corresponding author.
E-mail address: xin.sun@pnnl.gov (X. Sun).
[1] These authors contributed equally.

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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 min respectively followed by air-cool, see heat treatment schedule illustrated in Fig. 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.

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

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 min. 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. Fig. 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 Fig. 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 Fig. 3, are used to guide the subsequent data processing of the indentation load-depth curves obtained from the nanoindentation batch runs with 15 × 15 indent arrays.
(2) Nanoindentation batch run with 15 × 15 indent array:
To capture the statistical variations of the individual phase stress-strain response, a 15 × 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 5 × 5 indentation array was