

Application of Nano-Indentation Test in Estimating Constituent Phase Properties for Microstructure-Based Modeling of Multiphase Steels

Guang Cheng, Kyoo Sil Choi, Xiaohua Hu, and Xin Sun
 Pacific Northwest National Laboratory

ABSTRACT

For multiphase advanced high strength steels (AHSS), the constituent phase properties play a crucial role in determining the overall mechanical behaviors. Therefore, it is important to accurately measure/estimate the constituent phase properties in the research of AHSS. In this study, a new nanoindentation-based inverse method that we developed was adopted in estimating the phase properties of a low alloy Quenching and Partitioning (Q&P) steel. A microstructure-based Finite Element (FE) model was also generated based on the Electron BackScatter Diffraction (EBSD) and Scanning Electron Microscopy (SEM) images of the Q&P steel. The phase properties estimated from nanoindentation were first compared with those estimated from in-situ High Energy X-Ray Diffraction (HEXRD) test and, then, employed in the generated FE model to examine whether they can be appropriately used as the input properties for the model. The results show that the estimated phase properties from the inverse method are similar to those from HEXRD, and that the Ultimate Tensile Strength (UTS) and Uniform Elongation (UE) predicted from the FE model based on the estimated phase properties are also similar to those of tensile experiment of the Q&P steel. Based on the results in this study, the nanoindentation-based inverse method appears to be a viable way in determining the phase properties of complex multiphase steels with submicrometer grain sizes.

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INTRODUCTION

Advanced High Strength Steels (AHSS) represent an important technology in efforts to lightweight vehicles for improved fuel-efficiency and occupant safety. Main difference between AHSS and conventional high strength steels is their microstructures. Conventional steels are single phase materials whereas AHSS are primarily multiphase materials having several different phases in it, depending on its chemical composition and heating process. It is well known that, along with the microstructure, the constituent phase properties play a crucial role in determining the overall mechanical behaviors of AHSS, including the strength and ductility [1, 2, 3]. Therefore, measurement of accurate constituent phase properties is important in the study of AHSS. Various experimental methods have been explored for measuring the constituent phase properties of multiphase materials, which include micro/nano-pillar compression test [4], nanoindentation test [5, 6, 7, 8], in-situ High Energy X-Ray Diffraction (HEXRD) test [9].

Recently, we developed a new nanoindentation-based inverse method to estimate the constituent phase properties of a 3rd generation AHSS [5], and showed that the nanoindentation test may have advantages over the other two methods for the study of multiphase steels with submicrometer microstructural features, considering the experimental difficulties inherent in those two methods. In this study, the new inverse method we developed was adopted in estimating the constituent phase properties of a low alloy Quenching and Partitioning (Q&P) steel to examine its applicability in the research

of AHSS. For this purpose, the constituent phase properties estimated from the nanoindentation test of the Q&P steel were first compared with those estimated based on HEXRD test and, then, employed as the input phase properties in the microstructure-based Finite Element (FE) modeling to examine their appropriateness. The applicability of nanoindentation test for the measurement of constituent phase properties of AHSS is finally evaluated based on the obtained results.

EXPERIMENTS

The material used in this study is a low alloy (0.3C-3Mn-1.6Si) Q&P steel generated with heat processing parameters listed in Table 1. This material is enriched with carbon and manganese to obtain the desired austenite volume fraction and stability, and the heat processing parameters include the temperature and time for the initial reheating, quenching and partitioning as listed in Table 1 [9, 10, 11].

Microstructure analysis has been first performed on the Q&P steel using scanning electron microscopy (SEM). Figure 1 shows a SEM picture obtained from an etched in-plane cross-section. As shown in the figure, the material has very small-size grains (<3 μm) and have quite complex microstructures, mainly composed of three different phases (i.e., austenite (A), tempered martensite (TM), untempered martensite (UT)). Here, among the raised features, the thin-film-like grains are the austenite and the relatively large grains can be the austenite, untempered martensite or the mixture of them (A/UT) whereas the unraised feature is basically the tempered martensite [5, 11].

Considering the small grain size of the material, nanoindentation test to a depth of 30nm has been performed on the unetched surface using a Berkovich indenter tip. A 20 by 20 indentation array with a spacing of 1.5 μm between indentations was employed to ensure that sufficient indents for each phase can be used in obtaining the average phase stress-strain curves. The load-depth curves were recorded for all the indentations. After indentation, the surface was etched to reveal the microstructure of the tested area. Figure 2 shows the overlay of the indentation locations on the SEM image of the etched tested area, which was used to identify the phases at the indent locations. Each indentation was categorized into one of three different regions (i.e., TM region, A/UT region, A/TM region). Due to the small size of film-like austenite grains in A/TM region compared to the indentation size, the indentation within A/TM could not be separately categorized into A or TM. The detailed information on the nanoindentation test on the multiphase steels including Q&P steel can be referred to Cheng et al. [5] and Taylor et al. [12].

Table 1. Heat processing parameters for the model Q&P steel samples [9, 10, 11].

Reheating Temps/Time($^{\circ}\text{C}/\text{s}$)	Quenching Temps/Time($^{\circ}\text{C}/\text{s}$)	Partitioning Temps/Time($^{\circ}\text{C}/\text{s}$)
820/120	180/10	400/100

Electron BackScatter Diffraction (EBSD) technique was also implemented for the Q&P steel for purpose of phase identification and volume fraction. SEM analysis was then performed on the same location where EBSD was performed. Figure 3 shows the grain orientation map, phase map from EBSD and the SEM picture for the location of EBSD. According to the phase map in Figure 3(b), the volume fraction of austenite phase is approximately 18~19 %. Note that the images of orientation map and phase map from EBSD were used together with the SEM image in generating the microstructure-based FE model of the Q&P steel, which will be discussed later [11].

EXTRACTION OF STRESS-STRAIN CURVES FROM NANOINDENTATION TEST

In this section, the new inverse method that we developed to estimate the stress-strain behaviors of the constituent phases of multi-phase materials [5] is briefly introduced.

In this proposed method, the material's elasto-plastic behavior was assumed to be described as [13]

$$\begin{aligned} \sigma &= E\varepsilon & \text{for } \sigma \leq \sigma_y \\ \sigma &= \sigma_y \left(1 + \frac{E}{\sigma_y} \varepsilon_p \right)^n & \text{for } \sigma \geq \sigma_y \end{aligned} \quad (1)$$

where E is elastic modulus, σ_y is the yield strength and n is the hardening exponent. ε_p is the nonlinear part of the total strain ε , accumulated beyond the yield strain ε_y , and defined as $\varepsilon_p = \varepsilon - \varepsilon_y$. With the assumed power law in Eq. (1), the values E , σ_y and n can determine the material's overall stress-strain behavior.

The stress-strain curve was extracted from the load-depth curve obtained from nanoindentation test. Figure 4 shows the flow chart of the proposed extraction method consisting of four steps. In Step 1, the elastic modulus and hardness are calculated by the Oliver-Pharr method [14,15]. In order to ensure solution uniqueness, in Step 2, the empirical relationship between nanohardness and yield strength reported by Rodríguez and Gutierrez [16] was introduced to calculate yield strength. This leaves hardening exponent the only variable left to be determined. The representative stress (i.e., $\sigma_{0.033}$) is then calculated in Step 3 where a correlation factor considering the indentation size effect and indenter bluntness effect is also proposed to convert the loading curvatures from nanoindentation to microindentation [13]. In Step 4, the hardening exponent is finally calculated from yield strength and representative stress. Detailed explanation on the procedure in the proposed inverse method is referred to Cheng et al. [5].

The examples of results obtained based on the developed inverse method are presented in Figure 5. Figure 5(a) shows two load-depth curves obtained from the nanoindentation test of the current Q&P steel. The two curves in the figure were obtained from A/UT region. It appears that, due to the large difference between the two curves, one is for the austenite and the other is for the untempered martensite. Figure 5(b) shows the two corresponding stress-strain curves determined by the proposed method with the determined material parameters listed. As the proposed inverse method in Figure 4 can be used to determine a unique stress-strain curve for a given nanoindentation load-depth curve, the average phase properties of a multiphase material can now be obtained by averaging the multiple stress-strain curves obtained from the corresponding nanoindentation load-depth curves.

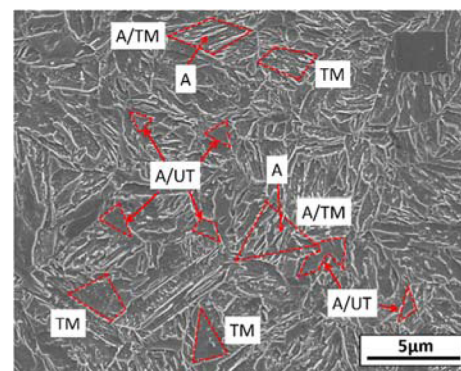


Figure 1. SEM micrograph of the Q&P steel (A, TM, A/UT and A/TM represent austenite, tempered martensite, mixture of austenite and untempered martensite and mixture of austenite and tempered martensite, respectively).



Figure 2. Overlay of indentation locations on SEM image of the etched Q&P steel.

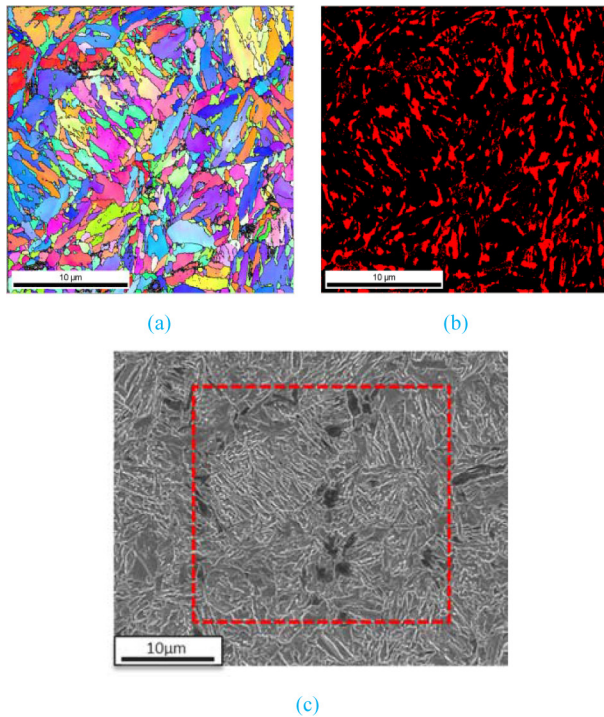


Figure 3. (a) Grain orientation map, (b) phase map from EBSD (red color: austenite) and (c) SEM micrograph of the same location following 2% nital etching. (The region in red-dashed box of Figure (c) corresponds to the EBSD location.) [11]

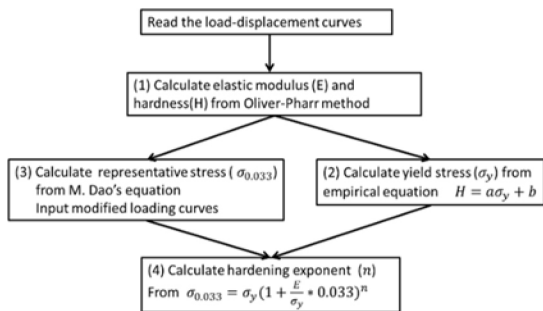
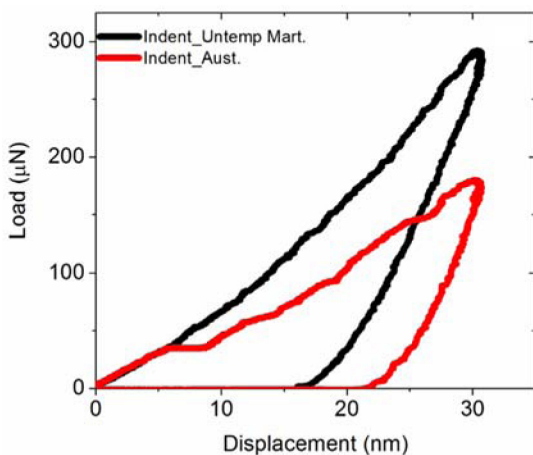


Figure 4. Algorithm of inverse calculation to estimate the elasto-plastic properties from indentation load-depth curve [5].



a.

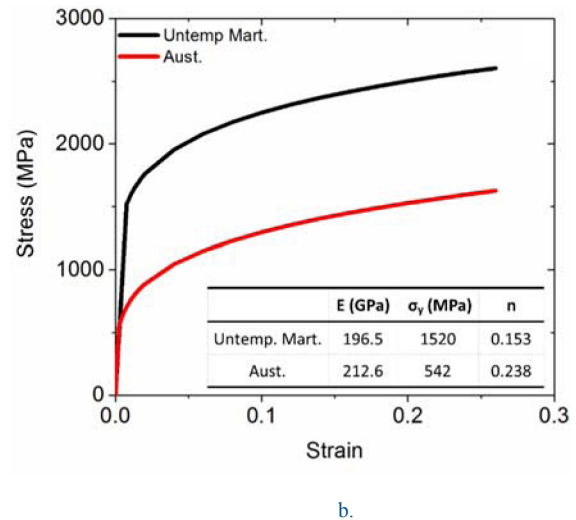


Figure 5. Example of inverse calculation: (a) Original load-depth curves and (b) stress-strain curves calculated from the proposed inverse method.

It is noted here that the deformation induced martensitic phase transformation from austenite does not appear to have been triggered during the nanoindentation test of the current Q&P steel since no evidence for phase transformation (e.g., rapid slope change in the loading curves) was observed from the indentation load-depth curves as in the Q&P steel in Cheng et al. [5]. Thus, martensitic phase transformation was not considered in this study.

DETERMINATION OF INDIVIDUAL PHASE PROPERTIES

Figure 6 shows the histograms of nanohardness values obtained for the Q&P steel, with the hardness distributions for TM, A/TM and A/UT regions in Figures 6(a), 6(b) and 6(c), respectively. The wide range of hardness values for each constituent region are observed as shown in the figure. As seen in the SEM in Figure 1(a), the grain size of TM is generally larger than the indentation size. Therefore, the hardness data for TM shown in Figure 6(a) can be considered to be solely from TM grains. However, the indentation within A/TM could not be separated for each constituent grain (i.e., A or TM) due to the small grain sizes in it compared to the indentation size. Hence, the hardness data shown in Figure 6(b) represents the combined hardness from austenite and tempered martensite in the A/TM. Although the grain size of A/UT is relatively large compared to the indentation size, the actual phase (i.e., A or UT) at the indentation location within the A/UT regions could not be identified from the simple observation of the SEM image shown in Figure 2. Note, however, that the hardness histogram of A/UT may approximate a bimodal distribution as schematically illustrated in Figure 6(c) due to the hardness difference between austenite and untempered martensite. It is assumed for the current Q&P steel that, among all the data for A/UT, the indentations with hardness lower than 6.8 GPa belong to austenite, whereas the indentations with hardness higher than 6.8 GPa belong to untempered martensite. Subsequently, 65 and 40 indents could be counted as austenite and untempered martensite, respectively.