

# Ultrahigh strength martensite–austenite dual-phase steels with ultrafine structure: The response to indentation experiments

R.D.K. Misra<sup>a,\*</sup>, P. Venkatsurya<sup>a</sup>, K.M. Wu<sup>b</sup>, L.P. Karjalainen<sup>c</sup>

<sup>a</sup> Laboratory for Excellence in Advanced Steel Research, Center for Structural and Functional Materials, University of Louisiana at Lafayette, P.O. Box 44130, Lafayette, LA 70504, USA

<sup>b</sup> International Research Institute for Steel Technology, Wuhan University of Science and Technology, Wuhan 430081, China

<sup>c</sup> Centre for Advanced Steels Research, University of Oulu, P.O. Box 4200, 90014 Oulu, Finland

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## ABSTRACT

In medium to high carbon steels, characterized by martensite–austenite microstructure processed by quenching and partitioning process, martensite potentially provides high strength, while austenite provides work hardening [Fu, Wu, and Misra, DOI: 10.1179/1743284712/068]. Given the significant interest in these steels in the steel community, the paper reports for the first time the nanoscale deformation experiments and accompanying microstructural evolution to obtain micromechanical insights into the deformation behavior of ultrahigh strength–high ductility dual-phase steels with significant retained austenite fraction of  $\sim 0.35$ . During deformation experiments with nanoindenter, dislocations were distributed on several slip systems, whereas strain-induced twinned martensite and twinning were the deformation mechanisms in carbon-enriched and thermally stabilized retained austenite. Furthermore, ultrafine dual-phase steels exhibited high strain rate sensitivity.

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## 1. Introduction

Martensite with medium to high carbon content is the phase that provides ultrahigh strength in steels. The background of high strength is related to the carbon content, and the strengthening mechanism seems to be the segregation of carbon to sub-boundaries [1] or formation of Fe–C nanocarbides or carbon-rich clusters [2]. However, the ductility of high carbon martensite is very low. The ductility can be improved by tempering of martensite or including a softer second phase in the microstructure, viz. retained austenite that transforms to martensite during plastic deformation. This increases the strain hardening, tensile strength and uniform elongation, if the stability of the austenite phase is convenient. An effective approach to stabilize austenite was originally proposed by Kinsman and Shyne in 1967 [3]. It was demonstrated that the diffusion of carbon from martensite to austenite retarded further transformation to martensite. Strong effects of stabilization took place when carbon diffusion from martensite led to build-up of carbon at the martensite/austenite interface. This carbon build-up during low temperature thermal treatment process, where carbon diffuses from martensite to austenite was referred as thermal stabilization [3]. In recent years, the thermal stabilization of austenite concept has evolved into the quenching and partitioning process (Q&P) [4–6]. In the

Q&P process, after austenitization, the steel is quenched between martensite start ( $M_s$ ) and martensite finish ( $M_f$ ) temperature, and held isothermally at the quench temperature or higher for an appropriate time to allow diffusion of carbon from the super-saturated martensite into the neighboring untransformed retained austenite before cooling to room temperature. To prevent carbide precipitation, alloying with Si and/or Al is considered. The volume fraction of retained austenite is dependent on the quenching temperature and carbon content of the steel. Increasing the carbon content increases the volume fraction of austenite and lowers the  $M_s$  (by  $\sim 450$  °C/wt% carbon).

It is recognized that the stability of retained austenite is crucial in terms of enhancing TRIP (transformation-induced plasticity) effect. It was observed that the different rates of austenite transformation can be attributed to the location, carbon content, and size of the retained austenite grains in the respective TRIP microstructures [7]. According to Jimenez-Molero et al. [8] both the carbon content and the grain volume play a key role in the stability of the small austenite grains, while the carbon content exerts the dominant effect in the stability of the big grains. This means that bulk granular austenite tends to transform readily to martensite whereas thin film-type austenite between martensite laths is more stable.

It is important to fundamentally understand the factors controlling ductility in high strength steels, and thereby the deformation mechanisms, especially in austenite, are of interest. Misra's group has employed nanoindentation to deform metastable austenitic alloys to investigate deformation mechanisms, martensite formation,

\* Corresponding author. Tel.: +1 337 482 6430; fax: +1 337 482 1220.  
E-mail address: [dmisra@louisiana.edu](mailto:dmisra@louisiana.edu) (R.D.K. Misra).

deformation twinning, and dislocation glide [9–11]. The aim is to probe the deformation processes in a small volume of material that can be considered to have a low probability of encountering pre-existing dislocations, at least prior to the commencement of plastic deformation and the tested volume is scalable with the microstructure. The objective of the present study is to explore nanoscale deformation behavior of retained austenite in ultrahigh strength dual-phase steels with the aim that it will help us elucidate a rational foundation concerning the processing of quenched and partition-tempered (Q-PT) type steels.

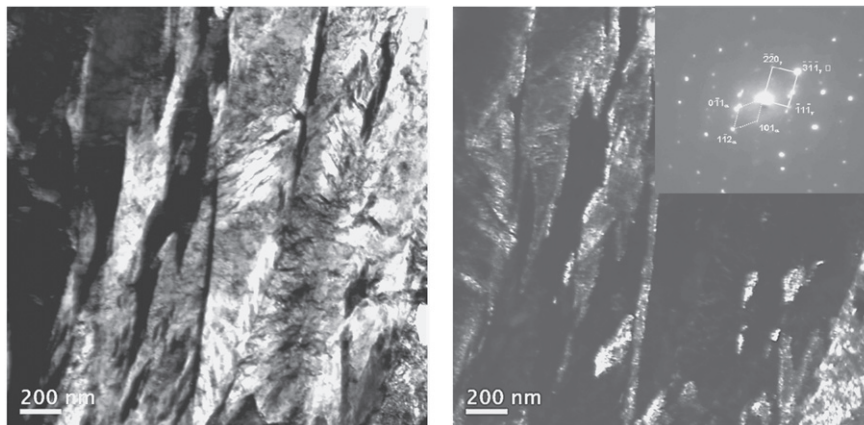
## 2. Materials and experimental procedure

The chemical composition of the starting material in weight % was Fe–0.95C–1.30Mn–0.91Si–0.23Cr. The steel contains appropriate silicon to suppress cementite formation and adequate manganese and chromium to provide hardenability. The alloy was homogenized at 1200 °C for 24 h in a vacuum annealing furnace, followed by furnace cooling to ambient temperature. For Q-PT treatment, specimens were austenitized at 1000 °C for 30 min and quenched in ice-water, and then partition-tempered (P-T) at 450 °C in a molten salt bath for 60 s and 300 s followed by water-quenching to room temperature. The results reported here are for 450 °C/60 s P-T condition to avoid the long term effect of precipitation of chromium and molybdenum carbides [12]. The martensite start ( $M_s$ ) and martensite finish temperature ( $M_f$ ) of the steel was calculated by JMatPro to be 80 °C and –15 °C, respectively.

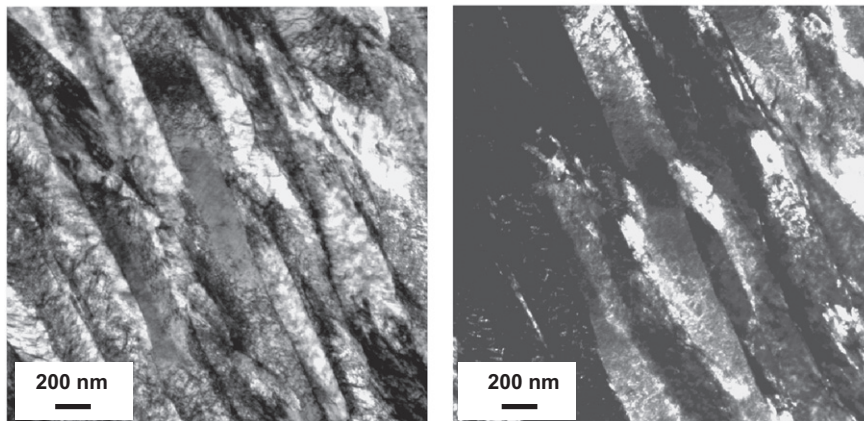
X-ray diffraction (XRD, Xpert Pro MPD) was used to determine the volume fraction of retained austenite and martensite, by integrating intensities of the (111), (200), (220) and (311) austenite peaks and (110), (200), (211) and (202) martensite peaks. The measurement error of retained austenite volume fraction was 0.015. The carbon concentration in austenite was estimated by using the measured lattice parameters [13]. The samples were step scanned in a X-ray diffractometer, operating at 40 kV voltage 45 mA current, using Cu-K $\alpha$  radiation. The  $2\theta$  scan angles ranged from 20° to 100°.

Nanoindentation experiments, as described previously [9–11], were carried out in load-controlled mode at a loading rate range of 2  $\mu$ N/s and maximum load set to 0.5 mN. The second set of experiments involved indentation at a constant strain rate, with the strain rate varied from 0.05 to 1 s<sup>-1</sup>, with the objective to study strain rate sensitivity of dual-phase steels with ultrafine structure. The nanoindenter system (MTS XP) consisted of a Berkovich three-sided pyramidal diamond indenter with a nominal angle of 65.3° and indenter tip radius of 20 nm. An array of indents of matrix 12  $\times$  12 was defined with the indent gap of 10  $\mu$ m. After the indentation experiments, the disk were removed from the mount and final electropolishing was carried out only from the side opposite to the indented surface. Using this procedure, the area surrounding the indents, which is present along the final jet-polished hole, was electron transparent to study the deformation behavior using a transmission electron microscope. The electron microscopy studies were carried out in the annulus surrounding the perforation, i.e. in the plastic zone. Indentations were made while looking through the microscope

### Example 1



### Example 2



**Fig. 1.** Representative bright-field (left) and dark-field (right) TEM micrographs of martensite–austenite dual-phase nanostructured steels. In the dark-field image, martensite is the dark-phase and austenite is the bright phase. Example 1, the selected area diffraction is also presented confirming the presence of austenite.

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