



## Reliability/unreliability of mixture rule in a low alloy ferrite–martensite dual phase steel



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### ABSTRACT

The aim of this paper is to investigate in details the relationship between the volume fractions of ferrite and martensite with the variation of hardness in a low alloy ferrite–martensite dual phase (DP) steel. For this purpose, a wide variety of ferrite–martensite DP samples consisting different volume fractions of ferrite and martensite have been developed using step quenching heat treatment cycle involving reheating at 860 °C for 60 min, soaking at 600 °C salt bath for various holding times followed by 70 °C hot oil quenching. Optical microscopy has been supplemented by electron microscopy and hardness measurements to follow microstructural changes and their relation to the variation in hardness. The results showed that there is a non-linear relationship between the hardness of DP samples with the volume fraction of phase constituents indicating that the mixture rule is not reliable in the ferrite–martensite DP microstructures. The unreliability of mixture rule is related to the variation of ferrite and martensite hardening responses developed in the DP samples. The DP microstructure consisting 6–7% volume fraction of continuous grain boundary ferrite in the vicinity of martensite has been associated with a remarkable higher hardness for both ferrite and martensite in comparison with the other DP microstructures. The higher martensite hardness is due to the higher carbon content of the remaining metastable austenite developed in the ferrite–austenite two phase field area, leading to the harder martensite formation on the subsequent 70 °C hot oil quenching. The harder ferrite grains have been developed as a consequence of more constraints induced in the ferrite grains during martensitic phase transformation. The higher martensite volume fraction in the vicinity of thinner continuous grain boundary ferrite networks has been associated with the harder ferrite formation.

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### 1. Introduction

Low alloy DP steels are one of the most important categories of engineering steels with concurrently good ductility, toughness and high strength. Some of the practical engineering applications such as automotive industry are still demanding commercial higher strength steels in conjunction with good ductility and toughness in order to lighten structural parts [1–5]. This unique combination of tensile strength and ductility has led to the emergence of a series of heat treated multi-phase low alloy steels in which the dominant microstructures are mainly characterized with about 20–25% of hard martensite islands in a soft ferrite matrix [6,7]. Most of the early researches have been restricted to the microstructures containing a low volume fraction of martensite in a polygonal ferrite matrix in order to obtain the optimum strength–ductility combination. This restriction relies on the fact that the mechanical properties of ferrite–martensite DP steels are mainly dictated by the volume fraction of martensite, an increasing in martensite volume

fraction has been associated to the more strength and of course simultaneously decreasing in ductility and impact properties.

In general, the strength and hardness of ferrite–martensite DP steels are not only related to the volume fraction of microphases but also to some other microstructural variables, e.g. prior austenite grain size, morphology and distribution of martensite, and the nature and amount of ferrite [4,8–10]. Two completely different models, e.g. a linear and non-linear relationship have been proposed between the hardness and martensite volume fraction in the low alloy ferrite–martensite DP steels [11–15]. The linear group claims that the strength and hardness of low carbon ferrite–martensite DP steels have been increased linearly with increasing the martensite volume fraction. On the other hand, the non-linear school proposes completely different models for the relationship between hardness and the volume fraction of microphases. These arguments are still underway and no agreement has been yet reached. Moreover, there have been quite contradicting reports even by the non-linear group, that probably the major differences can be due to the investigated steel compositions. Therefore, the purpose of this paper is to investigate in de-

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tails the relationship between hardness and volume fractions of ferrite and martensite in a low alloy ferrite–martensite DP steel.

## 2. Materials and experimental procedure

The steel used in the present investigation was a commercially low alloy variant of AISI4140 steel with the chemical composition shown in Table 1. All of the proposed heat treated samples were cut from the as-received 20 mm diameter steel rod and were first normalized after being reheated at 860 °C for 60 min. Then, the proposed DP samples were subjected to the heat treatment cycles in order to achieve various ferrite–martensite structures. For each DP specimen, the heat treatment process is consisted of the following sequential stages: (a) reheating at 860 °C for 60 min to get full austenite; (b) soaking in a 600 °C salt bath for 20–55 s to achieve various ferrite morphology and volume fraction; and (c) followed by 70 °C hot oil quenching (OQ) to transform the remaining metastable austenite to martensite. The heat treatment cycle is shown schematically in Fig. 1.

The polished specimens were prepared based on the ASTM E3 standard and were etched using a 2% nital etchant solution in order to reveal general microstructural observation under optical microscopy [16]. The volume fractions of ferrite and martensite were measured using point count method according to the ASTM E562-02 standard [17]. The microstructural characterization was also examined under scanning electron microscope (SEM) model TESCAN-VEGA-II operated at an accelerated voltage of 15 kV. For comparing the level of carbon partitioning in ferrite and martensite, an analysis of chemical composition was carried out at various locations within ferrite and martensite area with energy dispersive X-ray spectroscopy (EDS).

Microhardness tests were conducted within ferrite and martensite using a load of 5 g for ferrite and 10 g for martensite being applied for 20 s duration loading time using a Future Tech microhardness tester machine model FM700, and the data were reported as Vickers hardness numbers (VHNs). The microhardness data were collected from at least five tests for each microphase. All of the specimen surfaces for nanoindentation tests were mechanically polished and subsequently electropolished to remove all of the damaged layers. The nanoindentation tests were carried out within ferrite and martensite using a CSM machine model NHTX S/N: 001-03119 with a Berkovich indenter. Analyses for the tip calibration and the calculation of hardness numbers were carried out based on the method used by Oliver and Pharr [18]. The peak load was 10.00 mN in association with the applied loading rate of 20 mN/min.

## 3. Results

### 3.1. Optical micrographs and hardness

Typical microstructural developments of ferrite formation in the DP samples isothermally transformed at 600 °C over a wide range of isothermal holding times are shown in Fig. 2. Figs. 2a and b show a mixture of continuous grain boundary ferrite (CGBF) and quasi-polygonal ferrite (QPF) in the vicinity of martensite for the DP samples obtained at 600 °C for 20 s and 25 s, respectively. Fig. 2c and d are light micrographs for the DP samples isothermally transformed at 600 °C for longer holding times of 30 s and 35 s, respectively. As being illustrated for keeping a longer isothermal holding time at 600 °C, the ferrite morphology has been significantly changed from continuous grain boundary to more polygonal/quasi-polygonal appearance. At a later stage of isothermal transformation on the subsequent holding time, the incoherent  $\gamma/\alpha$  interfaces have been grown into the prior austenite grains and subsequently the growth of a grain boundary ferrite allotriomorph into the coherent side has been occurred. Therefore, the DP samples obtained at 600 °C for longer isothermal holding times of 45 s and 55 s are in association with the formation of more grain boundary ferrite allotriomorph which has been generally faceted in appearance consistent with the quasi-polygonal ferrite formation as indicated in the light micrographs presented in Fig. 2e and f, respectively. These observations indicate that as the isothermal holding time has been increased for the DP samples, the ferrite

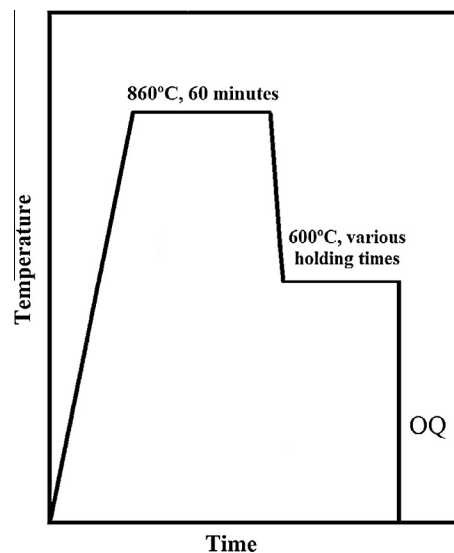


Fig. 1. Schematic diagram indicating the heat treatment cycle used to achieve various ferrite–martensite DP microstructures.

interfaces have been crossed over the prior austenite grain area and so the DP microstructures are characterized by more polygonal/quasi-polygonal ferrite formation. This ferrite growth mode can be rationalized due to the fact that the growth of polygonal ferrite is controlled by diffusional rearrangement of substitutional alloying elements along the coherent  $\gamma/\alpha$  interfaces and the long range diffusion of carbon atoms have been occurred from the growing ferrite interfaces [19]. Longer isothermal holding times could have facilitated these atomic diffusions and consequently more polygonal type ferrite has been formed in the microstructures.

Fig. 3 illustrates the variation of ferrite and martensite microhardnesses in association with microstructural changes developed in the DP specimens obtained at 600 °C for various holding times. The unique microstructure associated with the highest value of ferrite and martensite microhardnesses is shown in Fig. 3a. This structure is characterized by continuous grain boundary ferrite formation in the vicinity of martensite developed in the DP specimen obtained at 600 °C for 20 s. As the isothermal holding time has been increased from 20 to 35 s, a mixture of polygonal/quasi-polygonal ferrite at the prior austenite grain boundaries has been increasingly common in association with a significant decreasing in the ferrite microhardness as being indicated in Fig. 3b and c. For keeping a longer isothermal holding time of 45 s at 600 °C, a moderate variation in ferrite morphology and ferrite microhardness has been occurred in comparison to that of 35 s holding specimen (Fig. 3d). These results indicate that ferrite crystals are finer and harder with keeping shorter isothermal holding times at 600 °C.

Table 2 summarizes the results of hardness in association with the variation of ferrite and martensite volume fractions in the DP specimens obtained at 600 °C for different holding times. The ferrite microhardness has been sharply decreased from 360 to 223HV5 g with increasing the ferrite volume fraction from 7% to 14%, respectively. A moderate decreasing in ferrite microhardness

Table 1  
Chemical composition of the investigated AISI 4140 steel (in wt%).

C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Fe
0.384	0.208	0.673	0.0093	0.0063	0.971	0.154	0.0161	0.0234	Balance

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