



# Low-cycle fatigue behavior and microstructural evolution in a low-carbon carbide-free bainitic steel



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## ABSTRACT

This paper deals with the cyclic deformation behavior and microstructural evolution in a low-carbon carbide-free bainitic steel with two different microstructures. Low-cycle fatigue tests were performed at room temperature at various strain amplitudes under total strain control. The variations of the amount of retained austenite and the substructures versus the number of fatigue cycles were evaluated by the X-ray diffraction technique and electron microscopy. Fatigue test results demonstrate that the two microstructures exhibit very similar cyclic stress responses, i.e. initial cyclic hardening followed by cyclic softening or by cyclic saturation and softening till failure, depending on the strain amplitude applied. Parametric studies of the microstructure–property relationship indicate that the major cause for the initial cyclic hardening is neither martensitic transformation nor increased dislocation density. Based on these results and considering the initial high density of dislocations, which are pre-existent and mobile in the starting microstructure and which are entangled, rearranged or annihilated with cycling, the mechanisms responsible for the initial cyclic hardening followed by softening are analyzed.

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## 1. Introduction

Carbide-free bainitic steels show an excellent combination of strength, ductility and toughness, thus satisfying the ever-increasing demands for advanced high strength steels in various applications, such as for making automotive components and railway rails or crossings [1–3]. These steels are composed of a multiphase microstructure of fine bainitic ferrite and metastable retained austenite (subsequently indicated as Ar) with or without martensite [4–7]. While the fine sizes of bainitic ferrite laths may ensure high strength, the metastable retained austenite plays an important role in increasing the strength and also ductility, due to the transformation-induced plasticity (TRIP) effect [4,8,9]. During monotonic tensile loading, strain-induced austenite-to-martensite transformation generates a sustained high work-hardening rate, which tends to postpone the localization of plastic deformation, hence increasing the uniform strain and tensile strength [10–12]. However, in spite of the large volume of research into the monotonic deformation behavior in carbide-free bainitic steels [4–7, 13,14], very few studies have been devoted to their cyclic deformation behavior and microstructural evolution during fatigue loading.

Several previous publications [15–17] reported cyclic hardening and softening phenomena in conventional polygonal ferrite-based TRIP steels; these steels differ from carbide-free bainitic steels but similarly contain metastable retained austenite. According to Hilditch et al. [15,

16], the conventional TRIP590 and TRIP980 steels exhibit initial cyclic hardening followed by cyclic softening till failure at various strain amplitudes. Nevertheless, in the TRIP780 steel, initial cyclic hardening also occurs and is followed by cyclic softening during further fatigue loading at higher strain amplitudes; however, at lower strain amplitudes, cyclic softening occurs from the very first cycle up to fatigue failure [15,16]. It was thought that the initial cyclic hardening is due to the multiplication and interactions of dislocations [15,16]. Furthermore, strain-induced martensitic transformation generated during fatigue loading was also considered to contribute to cyclic hardening and even secondary cyclic hardening [15–19]. In contrast, the formation and spreading of dislocation sources or the recovery processes of dislocations were assumed to be the major cause for the initial cyclic softening, whereas the cyclic softening after the initial cyclic hardening was presumed to be due to the decreased dislocation density and the rearrangement of dislocations [15–17]. From the brief review of literature above, it is known that the conventional polygonal ferrite-based TRIP steels exhibit a variety of cyclic stress responses, which depend on the strength grade and strain amplitude applied.

The purpose of the present paper was to investigate and understand the cyclic hardening/softening behavior in a low-carbon carbide-free bainitic steel by means of low-cycle fatigue (LCF) test. Note that the conventional TRIP steels are characterized by the presence of polygonal ferrite as a major phase, which is soft and contains a relatively low density of dislocations. Conversely, fine-scaled, hard bainitic ferrite laths with extremely high densities of dislocations are the major constituent of carbide-free bainitic steels. Therefore, the retained austenite in

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carbide-free bainitic steels is expected to experience much stronger constraint from the surrounding hard bainitic laths. And such enhanced constraint may suppress martensitic transformation [9,20] and affect the evolution of dislocation substructures during fatigue loading [15–17]. In this paper, the changes of the amount of retained austenite and the evolution of dislocation density and substructure in the investigated carbide-free bainitic steel as a function of the number of fatigue cycles were measured by means of the X-ray diffraction technique and electron microscopy, attempting to clarify how the microstructure, especially the metastable retained austenite, evolves during fatigue loading. Finally, parametric studies based on the microstructure–property relationship were conducted to correlate the cyclic hardening/softening behavior with the evolved microstructure.

## 2. Materials and methods

The chemical composition of the steel is shown in Table 1. The bainite and martensite transformation starting temperatures were estimated to be 486 and 283 °C, respectively, by the JMatPro soft package. The steel was melt in a 25-kg vacuum induction furnace. The ingot was hot-forged into square bars of 20 × 40 mm<sup>2</sup> in cross section at a finishing temperature of 920 °C. Specimens were all austenitized at 920 °C for 30 min. After austenitization, the specimens were immediately austempered in a molten salt bath at a temperature of 300 °C for 3 h or at 350 °C for 1 h, before air cooling to room temperature, hereafter, referred to as 300- and 350-specimens, respectively. The choice of the two different austempering temperatures was to obtain two different amounts of retained austenite, and the holding times were chosen to ensure the finishing of bainitic transformation according to the JMatPro soft package and as indicated in Ref. [7].

Microstructures were examined by an optical microscope (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Specimens for OM and SEM observations were mechanically polished and chemically etched with a 2% Nital solution. Specimens for TEM observations were prepared by cutting samples into 400 μm thick slices before mechanical grinding down to 30 μm in thickness. Disks of 3 mm in diameter were twin-jet electropolished at room temperature to perforation. The perforated foils were examined using TEM at a 200 kV operating voltage.

Tensile properties were measured using plate specimens of 3 × 10 mm<sup>2</sup> in cross section and 20 mm in gage length. Tensile tests were performed at room temperature at a crosshead speed of 3 mm/min. Total strain-controlled tension–compression LCF tests were performed at room temperature at a strain rate of 6 × 10<sup>−3</sup> s<sup>−1</sup> using a 100 kN MTS test machine. Plate specimens of 3 × 4 mm<sup>2</sup> in cross section and 10 mm in gage length were used. The total strain amplitude (Δε<sub>t</sub>/2) ranging from 0.005 to 0.0125 was applied, using a triangular waveform and a strain ratio of R = −1. An axial extensometer with a gage length of 10 mm was used to measure the strain. The failure of specimens was determined by a reduction of 20% in peak tensile load with respect to the maximum peak value. Furthermore, for examining the microstructures evolved during fatigue loading, additional interrupted tests were performed at two strain amplitudes of 0.0055 and 0.012, by stopping the tests at typical numbers of cycles, such as after the tensile portion of the first cycle, at the maximum peak cyclic stress and at the half lifetime.

Deformed substructures and dislocation configurations at various fatigue states were examined by TEM, and the fracture surfaces of fatigue-failed specimens were observed by SEM. X-ray diffraction (XRD) analysis was used for the identification and quantification of

retained austenite in the as-austempered and fatigued specimens. Samples for XRD analysis were taken from the middle part of the gage section of cyclically-deformed specimens, or from locations about 3 mm away from the broken face of failed specimens. All samples were mechanically polished carefully and a thin surface layer was removed by chemical etching to avoid machining effects. XRD tests were performed using a D/max-2500/PC X-ray diffractometer with unfiltered Cu K<sub>α</sub> radiation at 40 kV and 200 mA. Carefully polished specimens were step scanned with a scan rate of 1°/min ranging from 20 to 120°. The volume fraction of retained austenite at various fatigue states was determined using the integrated intensities of (200)<sub>γ</sub>, (220)<sub>γ</sub> and (311)<sub>γ</sub> peaks of austenite and (200)<sub>α</sub>, (211)<sub>α</sub> and (220)<sub>α</sub> peaks of ferrite [21,22]. The carbon concentration in retained austenite (C<sub>γ</sub>, wt.%) was estimated from the austenite lattice parameter using the following equation [23]:

$$a_{\gamma} = 3.5780 + 0.0330C_{\gamma} + 0.00095Mn_{\gamma} + 0.0006Cr_{\gamma} - 0.0002Ni_{\gamma} + 0.0031Mo_{\gamma} + 0.0056Al_{\gamma} \quad (1)$$

where  $a_{\gamma}$  is the austenite lattice parameter in nm calculated with the Nelson–Riley extrapolation method, and  $Mn_{\gamma}$ ,  $Cr_{\gamma}$ ,  $Ni_{\gamma}$ ,  $Mo_{\gamma}$  and  $Al_{\gamma}$  represent concentrations of individual alloying elements in wt.%, assumed here to be the average contents in the steel.

Quantitative analyses of dislocation densities in the as-austempered and fatigued specimens were performed using the X-ray diffraction data. As is well-known, the microstrain can be determined from the broadening of diffraction profiles. And thus, the dislocation density,  $\rho$ , can be estimated according to the relationship between the microstrain and the square root of dislocation density as follows [24,25]:

$$\rho = \frac{K\varepsilon^2}{Fb^2} = \frac{6\pi\varepsilon^2}{b^2} \quad (2)$$

where  $F = 1$  and  $K = 12A$  ( $A = \pi/2$ ) [24,25],  $\varepsilon$  stands for the microstrain, and  $b$  is the Burgers vector of dislocations in  $\alpha$ -Fe.

## 3. Results

### 3.1. Microstructural characterization

Typical OM micrographs of the 300- and 350-specimens are shown in Fig. 1(a) and (b), respectively. Clearly, the two specimens show a very similar microstructure composed of fine acicular carbide-free bainitic ferrite (dark region) and retained austenite (light region) that is distributed between the sheaves of bainitic ferrite. SEM images are shown in Fig. 1(c) and (d), demonstrating more clearly the blocks of retained austenite and laths of bainitic ferrite. TEM microstructures are shown in Fig. 1(e) and (f), showing that the substructures in bainitic sheaves consist mainly of fine laths of bainitic ferrite and thin films of retained austenite that are located between the laths. The average thickness,  $t_B$ , of the bainitic ferrite laths was estimated using the following equation [4,13]:

$$t_B = \frac{2\bar{L}_t}{\pi} \quad (3)$$

where  $\bar{L}_t$  is the mean linear intercept length measured in a direction normal to the long edges of the laths. Thus, the thicknesses of bainitic ferrite laths in the specimens austempered at 300 and 350 °C are estimated to be 118 ± 22 and 156 ± 28 nm, respectively, indicating that the bainitic ferrite laths are obviously refined with decreasing the austempering temperature (Table 2). This refinement of bainitic laths is associated with the increased driving force for bainitic nucleation and thus increased bainitic nucleation rate with decreasing the isothermal temperature. Additionally, high densities of dislocations are observed in both bainite and austenite phases in the two specimens.

**Table 1**  
Chemical composition of the investigated steel (wt.%).

C	Si	Mn	Cr	Ni	Mo	Al	P	Fe
0.28	0.67	1.96	1.62	0.34	0.23	1.19	0.008	Balance

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