



## On the role of microstructure in governing the fatigue behaviour of nanostructured bainitic steels

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### ABSTRACT

Nanostructured bainite is not a novel laboratory-scale steel anymore and the interest on the commercial production of these microstructures by steelmakers and end-users is now conceivable. These microstructures are achieved through the isothermal transformation of high-carbon high-silicon steels at low temperature, leading to nanoscale plates of ferrite with thickness of 20–40 nm and retained austenite. Nanostructured bainitic steels present the highest strength/toughness combinations ever recorded in bainitic steels (2.2 GPa/40 MPa m<sup>1/2</sup>) and the potential for engineering components is alluring. However, fatigue properties, responsible of the durability of a component, remain to be examined. In order to understand the role of the microstructure during the fatigue crack propagation, the crack path in three nanoscale bainitic structures has been analysed on the basis of the relationships between grain misorientations and grain boundaries by Electron Backscatter Diffraction. Active slip systems in bainitic ferrite and crack deflection at grain boundaries have been identified, while retained austenite is cast doubt on its role.

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### 1. Introduction

Since the development of nanostructured bainitic steels [1–5], steel makers, end-users and scientists have devoted great attention and efforts to understanding both the transformation mechanism [6–15] and the origin of their outstanding mechanical properties [16–24] towards their industrialization [25]. It is well established that the nanoscale ferritic plates achieved through slow transformation at very low temperatures are responsible for strengthening up to levels of 2.2 GPa with a hardness of about 700 HV [5]. These plates of bainitic ferrite are dispersed in carbon-enriched regions of retained austenite, which is assumed to be determining for ductility [21,24,26], though the deformation mechanisms responsible for such an advantageous combination of properties are the subject of ongoing discussions.

Nonetheless, most applications of structural materials involve cyclic loading and life of a component is generally limited by fatigue and not by static strength. In order to fully exploit the potential of

nanostructured bainitic steels in industrial applications, a better understanding must be achieved on the relationship between microstructure and in-service properties.

As of today, little work has been published on nanostructured bainite fatigue properties. Peet et al. were the first authors on reporting fatigue tests on these steels [27]; with a stress ratio  $R=0.1$  they set the maximum number of cycles on  $10^5$  and extrapolated the results up to  $10^7$  cycles to estimate the fatigue strength. The validity of this method is undermined when considering the anomalous two-stepwise or duplex  $S-N$  curves observed in high-strength steels [28–33]. Later results on high-cycle bending fatigue on nanostructured bainitic steels [34] revealed that the fatigue limit for no failure in  $10^7$  cycles is determined not only by hardness or strength but also by microstructure, so that retained austenite and secondary cracks in fatigue fractures have positive effects on the fatigue resistance. Latest work of Leiro et al. [35] comparing the rotating bending fatigue behaviour of Quenched and Tempered (QT) and nanobainitic steels, showed an increase in fatigue properties of the latter mainly as a result of the refinement of the microstructure, the suppression of carbide precipitation and the Transformation Induced Plasticity (TRIP) effect. Regarding the latter, the benefits of the retained austenite in the fatigue performance of bainitic steels have been thoroughly discussed [27,34,36]. It has been found that the presence of retained austenite increases the threshold intensity

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[36], and its stress or strain induced transformation to martensite can blunt the crack by absorbing more energy than necessary for fatigue crack propagation [34].

On the other hand, many engineering components contain a variety of stress concentrators such as grooves, fillets, holes, or non-metallic inclusions. It has been observed that fatigue failure usually occurs as a result of crack initiation and growth from these stress raisers. Then, correct estimations of stress/strain concentration and crack development in the critical region are essential for practical machine design in service loading. Hence, in this work notched tension-tension fatigue will be explored in order to study the behaviour of nanostructured bainitic microstructures for actual engineering applications.

The fatigue limit of most metallic materials, for both notched and un-notched bodies, is determined by the propagation condition of nucleated small cracks. The crack closure and microstructural barriers are the two mechanisms for blocking small crack propagation at the fatigue limit [37]. Microstructural barriers in bainitic steels come from the nature of the displacive transformation itself, where it is generally assumed that close-packed {111} plane of the austenite (face-centred cubic, FCC) is exactly parallel to a {110} plane of the bainitic ferrite (body-centered cubic, BCC). In nanobainitic steels, most of the bainitic plates and the parent austenite interfaces are found to have an orientation relationships (OR) close to the Nishiyama–Wassermann (N–W),  $\{111\}_{\parallel}\{110\}_{\alpha}$  with  $\langle 110 \rangle_{\parallel}\langle 001 \rangle_{\alpha}$  [38,39]. The transformed products, however, possess the lattice invariant line rather than the plane and direction parallelism between bainitic ferrite and parent austenite. Therefore, the relative orientation can never be precisely the N–W, and it may alter between Kurdjumov–Sachs (K–S) and N–W depending in the ratio of the lattice parameters between the parent austenite and bainitic ferrite [40]. From a single austenite crystal, 12 crystallographic variants can be formed with a N–W orientation relationship due to the symmetry of cubic systems. A crystallographic packet is defined as a group of crystallographic variants with common {111} austenite plane (i.e. the same habit plane). Each bainite packet can be divided into bainite blocks of the three variants of the N–W relationship satisfying the same parallel plane relationship [41]. In consequence, the microstructure obtained is highly misoriented featuring a wide variety of potential microstructural barriers.

This work will analyze the fatigue crack propagation in nanostructured bainitic steels on the basis of its deflection due to the crystallographic orientation and boundary distribution. The main goal is to understand the role of both ferrite and austenite crystals on crack transmission and deflection across boundaries, and how this finally has an effect on fatigue life.

## 2. Materials and experimental procedure

The chemical compositions of the alloys investigated are 0.67C–1.67Si–1.31Mn–0.20Ni–1.73Cr–0.15Mo–0.18Cu–0.03Al–0.12V and 0.9C–2.47Si–0.74Mn–0.12Ni–0.97Cr–0.03Mo–0.17Cu–0.02Alwt%, encoded as 0.6CV and 1CSi, respectively. Heats were industrially manufactured via electric arc furnaces. Once solidified, the ingots were reheated to 1200 °C and hot rolled to 120 mm diameter bars for 0.6CV steel and to 35 mm bars for 1CSi steel, which were afterwards slowly cooled down in a furnace to avoid cracking. After cooling, the bars were annealed for 2 h at 690 or 700 °C to further soften the material and ease machining of mechanical testing specimens.

Tensile and fatigue specimens were pre-machined after annealing to within 0.3 mm of the final dimensions and then austenitized. Austenitization conditions were selected in a way to obtain the same prior austenite grain size in all the specimens. The following three different nanobainitic microstructures were obtained after subsequent isothermal treatments: 0.6CV at 250 °C during 10 h, 0.6CV/250; 0.6CV at 270 °C during 7 h, 0.6CV/270; and 1CSi at 250 °C during

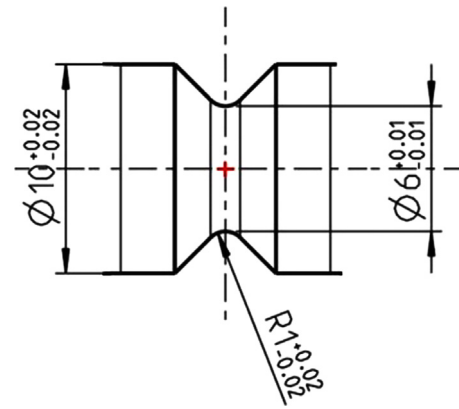


Fig. 1. Notched geometry used for tension-tension fatigue tests.

16 h, 1CSi/250. After transformation, the specimens were hard machined to the final dimensions under identical conditions to achieve similar surface roughness.

Tensile tests were conducted at room temperature and a strain rate of  $0.004 \text{ s}^{-1}$  with round tensile specimen with a gauge diameter of 8 mm and gauge length of 20 mm.

Notched conditions selected for fatigue testing are representative of the functioning of heavily loaded diesel injection components. The geometry of the notched specimen is shown in Fig. 1. Fatigue tests were carried out in a pulsating machine following the Up-and-Down strategy [42] at a stress ratio ( $R$ ) of 0.1 and a loading frequency of 150 Hz. Fatigue strength for a probability of failure of 50% (mean value) at  $10^7$  cycles was calculated according to the Probit method [42]. Failure mechanisms were distinguished by Scanning Electron Microscopy (SEM) examination of the fracture surfaces after testing. Microstructure below the fatigue fracture surface was examined by Field Emission Gun – Scanning Electron Microscopy (FEG–SEM) and Electron Backscatter Diffraction (EBSD) on nickel-plated cross sections of fracture surfaces cut through the crack initiation point on samples for 0.6CV/250 and 0.6/270 and 1CSi/250.

Samples for metallographic observation, both after fatigue testing and in the undeformed condition, were cut, ground and polished down to 2  $\mu\text{m}$  diamond paste following the standard procedures. A 2% Nital etching solution was used to reveal the microstructure going over several cycles of etching and polishing to favour the etchant penetration, especially relevant for bainitic nanostructures. SEM observation was performed on a FEG–SEM HITACHI S–4800 field emission gun scanning electron microscope, operating at 7 kV. In the case of the nanostructured bainite samples, high magnification FEG–SEM micrographs were used to determine the distribution and size of austenite blocks and coarse films, as well as the bainitic ferrite plate thickness,  $t$ , by measuring the mean lineal intercept  $L_T = \pi t/2$  in a direction normal to the plate length [3,43].

Quantitative XRD analysis was employed to determine the fraction of retained austenite and its lattice parameter for the estimation of the average carbon content in solid solution. For these experiments, samples were machined, ground and polished with 1  $\mu\text{m}$  diamond paste, and then subjected to several cycles of etching and polishing to obtain an undeformed surface; finally the samples were polished with colloidal silica. X-ray diffraction measurements were performed by means of a Bruker AXS D8 diffractometer equipped with a Co X-ray tube and Goebel mirror optics to obtain a parallel and monochromatic X-ray beam. Operational parameters and the procedure for obtaining the austenite content and composition are described elsewhere [21,44,45].

Specimens for EBSD observations were carefully polished with 50 nm colloidal silica suspension at the final stage of metallographic preparation. EBSD analysis was performed by means of the HKL CHANNEL 5 system (Oxford Instruments) coupled to the FEG–SEM JEOL JSM 6500F microscope, operating at 20KV. A  $16.5 \mu\text{m} \times 13.5 \mu\text{m}$

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