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Hélène Bultel, Jean-Bernard Vogt. Influence of heat treatment on fatigue behaviour of 4130 AISI steel. *Procedia Engineering*, 2010, 2, pp.917-924. 10.1016/j.proeng.2010.03.099 . hal-02512387

**HAL Id: hal-02512387**

**<https://hal.univ-lille.fr/hal-02512387v1>**

Submitted on 19 Mar 2020

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 ScienceDirect

Procedia Engineering 2 (2010) 917–924

Procedia  
Engineering

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Fatigue 2010

## Influence of heat treatment on fatigue behaviour of 4130 AISI steel

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Received 8 March 2010; revised 11 March 2010; accepted 15 March 2010

### Abstract

The 4130 steel is widely used in petroleum and gas industry. During the use, it can be exposed to high temperatures for long duration as well as to severe cyclic loading as a consequence of start and shut down procedure. Both have an effect on microstructure and on mechanical strength, especially fatigue resistance. The goal of this study is to characterize the influence of a thermal treatment on high temperature fatigue behaviour of this steel.

The material has been investigated in its as-received condition and after a high temperature heat treatment (around 1000°C and slowly cooled down in calm air). The microstructure changes from a bainitic to a ferrito-pearlitic one.

The fatigue tests are conducted at 450°C under total strain control ranging from  $\Delta\varepsilon_1 = 0.8\%$  to 1.5%. It is shown that the ferrito pearlitic steel exhibits a primary and secondary hardening while the bainitic one is ore stable after the initial hardening. The fatigue resistance is better for the ferrito pearlitic steel than for the bainitic steel when plastic strain variation is considered but the conclusion inverses with the cyclic stress amplitude. Crack nucleation as well as crack growth were found to be promoted in the bainitic steel. © 2010 Published by Elsevier Ltd. Open access under [CC BY-NC-ND license](http://creativecommons.org/licenses/by-nc-nd/3.0/).

*Keywords:* secondary hardening; short cracks; ferrito-pearlitic and bainitic steel

### 1. Introduction

It is well known that the thermal history of steels strongly influences their microstructure and their mechanical properties. Even when the material has received its recommended heat treatment before use, the in-service conditions can act as a heat treatment. This is a typical situation for components of power plants submitted to high temperatures and stresses for very long term duration. For instance, long term exposure (in the range 160 000h) of 2¼Cr1Mo ferrito-bainitic steels affect the stability of precipitates as well as dislocations densities in both grain types, and afterwards the fatigues behaviour [1]. Another kind of unwanted but unavoidable heat treatment is the coating by thermo-mechanical heat treatment of a material.

In this study, attention is focused on fatigue behaviour of AISI 4130 steel. This steel is a low alloy steel containing molybdenum and chromium as strengthening agents and is widely used in structural applications such as aircraft engine mounts or welded tubing applications. It can also be exposed to long term stays at high temperatures. Several studies have been devoted to describe the fatigue behaviour of this steel. However, depending on the heat treatment, even if conventional, the microstructure is different, being sometimes ferrito-pearlitic [2, 3], or tempered

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martensitic [4] or even bainitic [5]. In the present study, the specific heat treatment applied to the 4130 steel results from the thermal cycle that occurs during thermo-mechanical heat treatment. Therefore, the fatigue behaviour of the 4130 steel will be studied in the as-received API norm condition and after a high temperature high pressure heat treatment.

## 2. Materials and experimental procedure

The chemical composition of the AISI 4130 steel used in this study, given in Table 1, follows the standard. It is prepared according to API6 NACE MR 01.75 oil specifications, and will be referred to the as-received condition. A special thermo-mechanical treatment is performed on the steel. It consists in an austenitization at 1155°C while applying 1000 bar pressure during 3 h. Then the material is very slowly cooled in the furnace. Their microstructure was revealed by optical microscopy (OM) using the Nital etchant (4% nitric acid 96% ethanol).

Table 1: composition of AISI 4130 S

C	Mn	Cr	Mo	P	S	Si	Ni	Cu	Fe
0.28-0.33	0.40-0.60	0.80-1.10	0.15-0.25	0.025	0.025	0.15-0.35	0.25	0.35	bal

The fatigue tests are carried out using a Schenk hydraulic machine with a load capacity of 250 kN under total axial strain control  $\Delta\varepsilon_t$  ranging from 0.8 to 1.2%. A fully push pull mode ( $R_e=-1$ ), a triangular wave form and a constant strain rate of  $4.10^{-3} \text{ s}^{-1}$  are used. All the tests are performed at 450°C in air using a radiation furnace. The temperature is checked thanks to a thermocouple rolled around the sample gauge length. The fatigue specimens are smooth and cylindrical with a gauge length of 10 mm and a gauge diameter of 6 mm. Their surfaces are finely polished in the solicitation direction before testing. The fatigue life is defined as the number of cycles leading to a drop of 25% of the tensile stress taking as a reference the mid-life pseudo stabilized hysteresis loop.

A FEI Quanta 400 scanning electron microscope (SEM) in the secondary electron and backscatter electron mode is used to analyze post mortem samples. After the tests and the surface examinations, the samples are longitudinally cut in order to analyze the origins, path and depth of the cracks. This was done by SEM after polishing and etching with Nital 4% which allows the cracks are qualitative and quantitative studies of the crack network.

## 3. Results

### 3.1. Heat treatment response

In the as received condition, the microstructure appears to be ferrite-bainitic with about 10% ferrite grains embedded in a continuous bainitic “matrix” ( Fig. 1a). The thermo-mechanical heat treatment leads to a ferrite-pearlitic microstructure containing about 60% ferrite and 40% pearlite ( Fig. 1b). In the rest of the text, the first microstructure will be referred as the bainitic one whereas the other one will be called the ferritic-pearlitic one. The grain size in the ferritic-pearlitic one is much higher than in the bainitic one.

The basic monotonic mechanical properties are collected in Table 2.

Table 2: Differences in basic mechanical properties between before and after heat treatment

Steel	Bainitic	Ferritic-pearlitic
Young Modulus GPa	200	197
Yield stress MPa	200	245
Vickers Hardness 50g	Bainite 300 – Ferrite 170	Ferrite 160 - Pearlite 220
Grain size ( $\mu\text{m}$ )	10	25

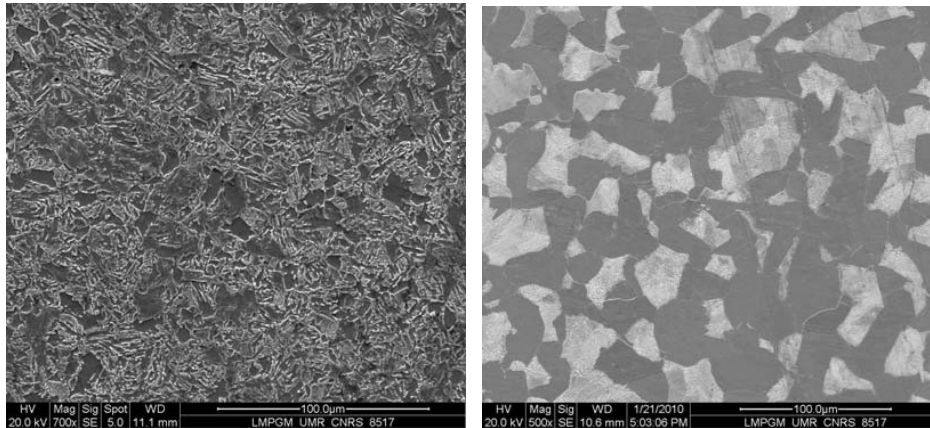


Fig. 1: SEM micrographs of the as received steel (left) and heat treated steel (right) showing respectively the bainite-ferrite microstructure and the ferrite-pearlitic microstructure.

### 3.2. Cyclic stress response

The evolution of the stress amplitude  $\Delta\sigma/2$  versus the number of cycles  $N$  is plotted in logarithmic scale in the Fig. 2 (a) for both the bainitic (open symbols) and the ferritic-pearlitic steels (closed symbols). It can be shown that the general behaviour in fatigue is relatively dependant on the microstructure. Under imposed strain, the response of both alloys is characterized by an initial cyclic hardening during the very first cycles and a secondary hardening period later in the fatigue life. These two hardening periods are connected by a transient period where the stress is more or less stables. The transient period is very dependant on the steel, being very short for the ferritic-pearlitic steel while rather long for the bainitic one. As well, the secondary hardening differs from one alloy to the other one by the hardening rate. For high strain tests, the transient period seems to include a weak but noticeable softening for the bainitic steel.

In Fig. 2 (b), the stress amplitude is plotted versus the fraction of life.

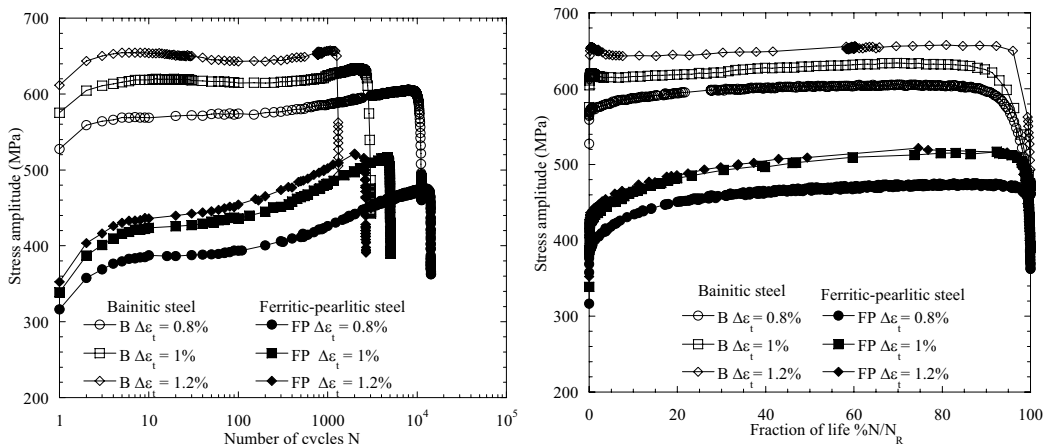


Fig. 2: Cyclic stress response as a function of the number of cycles  $N$  (a) and the life fraction (b) for various strain range tests at 450°C in air for the bainitic and the ferritic-pearlitic 4130.

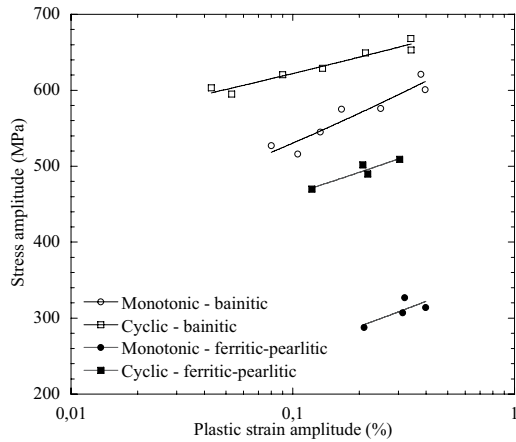


Fig. 3: Monotonic and cyclic stress-strain curves

It is confirmed that the hardening period of the ferritic-pearlitic steel occupies the whole fatigue life, whereas the bainitic steel appears to be more stable during the main part of the material life. The final decrease of the stress which is associated with crack propagation in the bulk seems also to depend on the steel. In the ferritic-pearlitic steel, it takes less than 5% of the fatigue life whereas in the bainitic one, it represents up to 10% of the sample life. In both cases, the primary hardening duration is negligible in terms of fraction of life as it only takes less of 2% of the fatigue life.

3.3. Monotonic and cyclic strain-stress curves

The monotonic and cyclic stress-strain curves are shown Fig. 3. The stress amplitude  $\sigma_a$  and the plastic strain amplitude  $\Delta\varepsilon_{pa}$  for the cyclic curves are measured on the mid-life hysteresis loop whereas the stress amplitude  $\sigma_{1/4}$  and the plastic strain amplitude  $\varepsilon_{p1/4}$  for the monotonic curves are taken from the first quarter of a cycle. These curves can be fit with the power law for monotonic (1) and cyclic (2) curves.

$$\sigma_{1/4} = K \varepsilon_{p1/4}^n \quad (1) \quad \text{and} \quad \sigma_a = K' \left( \frac{\Delta\varepsilon_{pa}}{2} \right)^{n'} \quad (2)$$

Where  $K$  is the monotonic strain hardening coefficient,  $n$  the monotonic strain hardening exponent,  $K'$  the cyclic strain hardening coefficient and  $n'$  the cyclic strain hardening exponent. The values of the coefficients and exponents are collected in Table 3.

Table 3: Coefficients of equations (1) and (2) for the steels

Steel	Bainitic	Ferritic-pearlitic
K	673.11	371.67
n	0.103	0.155
K'	696.91	565.82
n'	0.049	.086

The cyclic and monotonic stress-strain curves allow us to appreciate the considerable hardening of both the steels by cyclic loading, especially for the ferritic-pearlitic steel.

3.4. Fatigue life

The relationship between plastic, total strain variations as well as stress amplitude and fatigue life is plotted Fig. 4. At first, the number of cycles to failure is quite the same for both steels at a same total strain range. When it comes to plastic strain range, the ferritic-pearlitic steel shows a better resistance. However, the situation is inverted for comparison in terms of stress amplitude versus number of cycles to failure where the bainitic steel appears to be more resistant than the other one.

3.5. Longitudinal cut observations

The observations on transversal cuts of the specimens show that short cracks can initiate as well in ferrite grain as in pearlite grains in the ferritic-pearlitic steel. For the bainitic one, it is difficult to state in favour of one or the other type of grain due to the low volume fraction of ferrite. Therefore, only short cracks were seen in both grains. The crack density is also different from bainitic steel to ferritic-pearlitic one. Crack partition was analysed regarding to their length.

Fig. 5 shows the repartition of the cracks versus their length for the test performed at  $\Delta\epsilon_t = 1.2\%$  for the ferritic-pearlitic steel and  $\Delta\epsilon_t = 1.5\%$  for the bainitic one in order to compare tests with quite the same plastic strain at mid life,  $\Delta\epsilon_p \approx 0,64\%$ . As the less deformed samples of both steels show very little fatigue cracks, similar study has not been possible at lower deformation. The crack density is much higher (nearly 5 times) in the bainitic steel than in the ferritic-pearlitic steel. In both alloys, there was a ratio of 2 between 20  $\mu\text{m}$  length cracks and 50-100 $\mu\text{m}$  length cracks. The micrographs of Fig. 6 show that after very short crack nucleation at the surface of the specimen, the crack path during bulk propagation occurs differently according the steel.

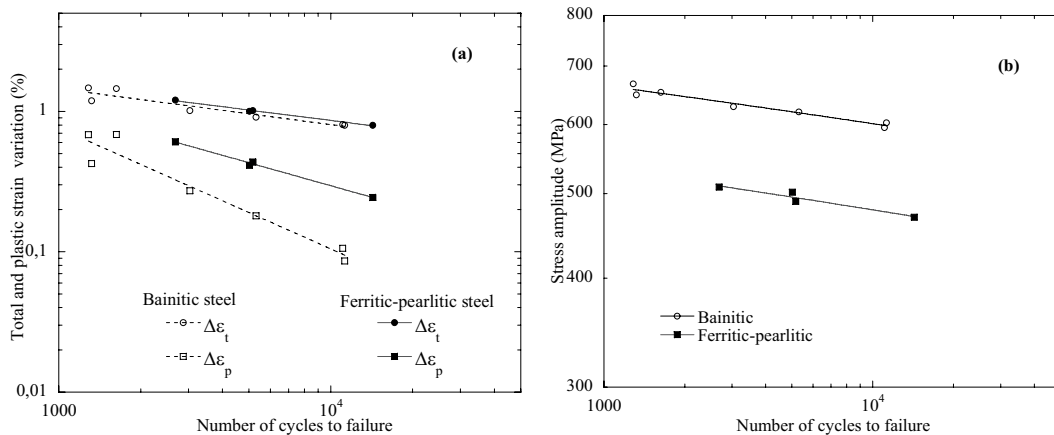


Fig. 4: Fatigue life as a function of plastic and total strain ranges (a) and as a function of stress amplitude (b)

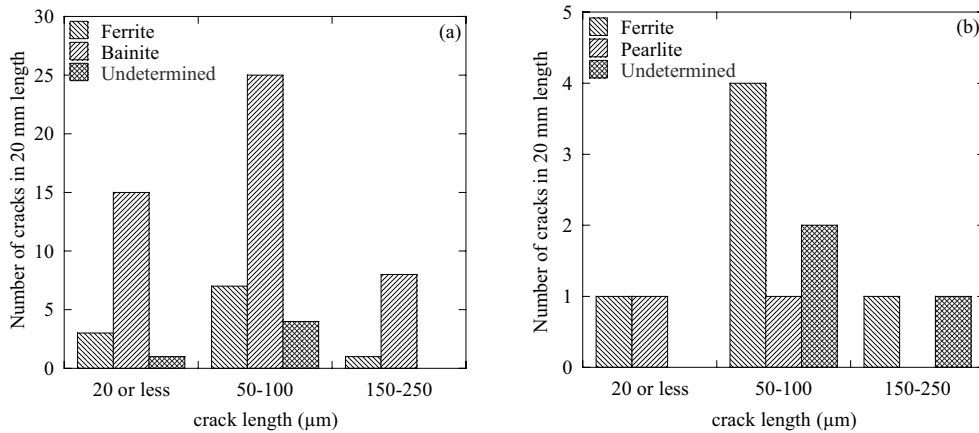


Fig. 5: Repartition of the fatigue cracks on post mortem samples after a fatigue test where the average plastic strain was  $\Delta\epsilon_p=0.64\%$  (a) bainitic steel (b) ferritic-pearlitic steel

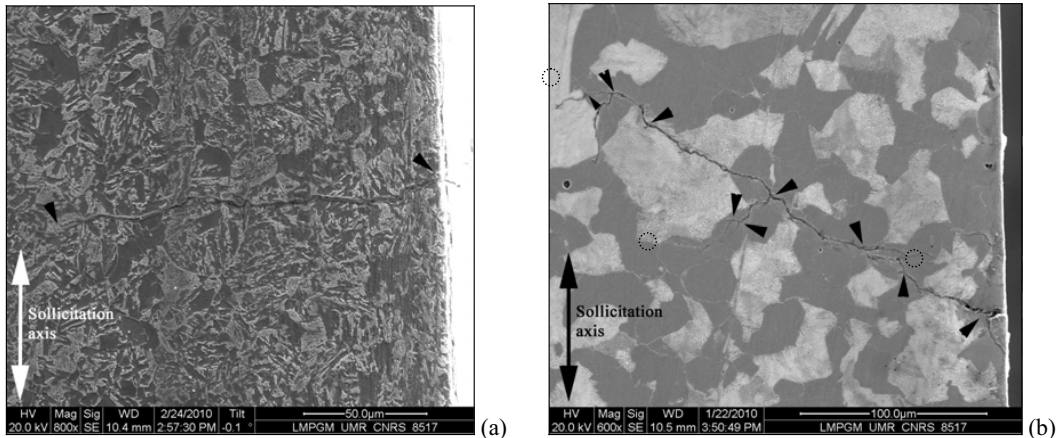


Fig. 6: Fatigue crack growth path of the two considered steels - (a) the bainitic one fatigued at  $\Delta\epsilon_i=1.5\%$  (b) the ferritic-pearlitic one fatigued at  $\Delta\epsilon_i=1.2\%$  – (the dark arrows show the crack branching, the dotted circles the crack tips arrests due to pearlite)

In the bainitic steel, crack advances transgranularly, rather perpendicular to the loading axis, and the crack morphology is nearly straight. In the ferritic-pearlitic steel, the average crack is inclined of about 45°-50° in regards with the loading axis. Crack path is more tortuous with obvious branching. There was an easier tendency to propagate in the ferrite matrix than in pearlitic areas.

#### 4. Discussion

The present study shows that a high temperature thermo-mechanical treatment leads to a deep change in the microstructure of the AISI 4130 steel. In terms of grain nature, the process exchanges bainite grains (present before thermo-mechanical treatment) for pearlite ones (after) while ferrite grains are found in both cases. In addition, it also changes the nature of the matrix which, respectively, was a bainitic one to a ferritic one, and the volume fraction of dispersed grains. This led us to distinguish and label two materials, the bainitic steel and the ferritic-pearlitic one. It is therefore reasonable to assign the stress to strain response with the evolution and behaviour of bainite grains and of ferrite ones.

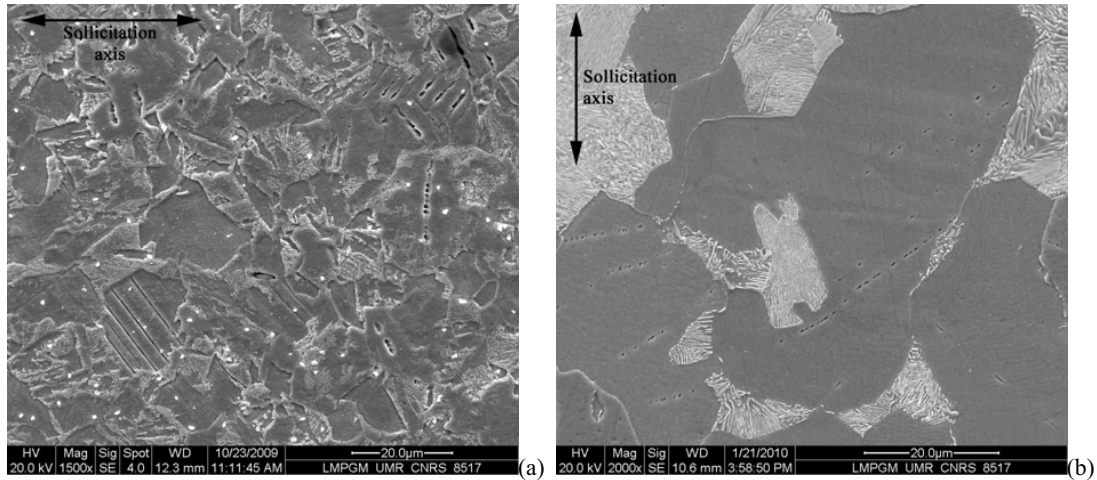


Fig. 7 : Ferrite accommodation of deformation in steel fatigued at  $\Delta\epsilon_f=0,8\%$  (a) bainitic steel (b) ferritic-pearlitic steel

In the ferritic-pearlitic steel, ferritic matrix grains are expected as a low dislocation density solid solution and the pearlite grain as alloyed cementite separated by rather wide ferrite lamellas. Hence, the initial hardening of the ferritic-pearlitic steel is unavoidable and attributed to the response of ferrite grains. It is reasonable to think that a cellular dislocations structure forms for these strain variations at high temperature as is observed in several pure or alloyed ferrite material [6,7]. The secondary hardening occurrence has also been reported in ferritic steels for some conditions [6,7,8] but not so much discussed, especially in this range of temperature and type of steel. Nevertheless, it has been shown [1,9] that the building of a dislocation structure in long term aged ferrite grains of a 2.25Cr1Mo could evolve through changes in fatigue cell misorientation. Though TEM investigation is needed to confirm this point, it is suspected that such a mechanism is responsible for the secondary hardening.

For the bainitic steel, the situation is different since bainite contains a network of dislocations resulting from the bainitic transformation which is however deformable due to the double tempering heat treatment. At the beginning of cycling, available dislocations are involved in the accommodation of cyclic plasticity in a similar way, as it occurs for recovered bainitic steel [1, 9], and results in an unavoidable softening. In bi-phased microstructures steels, a load transfer occurs from a softening active grain to an inactive one, both different in nature [9, 10]. Even in low volume fraction, the presence of soft grains plays a role in the load transfer [11]. Coupling with the continuous softening of the bainite matrix to the progressive hardening of ferrite grains could explain why the bainitic steel remains quite stable during the main part of his fatigue life. SEM observations of metallographic transversal cuts of fatigue specimen account for this difference for cyclic accommodation mechanisms between these two steels (Fig. 7) where bands assigned to intensive deformation or localized marks are observed in the bainitic steels.

As result of the load transfer process and localized strain in the newly activated grains, crack initiation can be expected in ferrite grains. However, as commented above, the fact that it was not so much observed may be due only the low volume fraction of ferrite grains. Nevertheless, that a crack initiate in ferrite grains or bainite grains, the propagation will guided by the lamellar texture of bainite grains. Indeed, if a crack is present in a ferrite grain, the most probable grain to be encountered is a bainite grain. On the other side, if bainite grain is cracking, it is very likely that the length will be much larger than the ferrite zone which could have played a shielding effect. This could explain why microcracks are perpendicularly oriented with the load axis.

In the pearlitic steel, ferrite being active most of the fatigue life, cracks unsurprisingly initiate and propagate in the ferrite grains. Tortuous cracking was more surprising to be observed because pearlite grains were rather homogeneously distributed in the ferrite matrix. Indeed, according to Korda et al [3,12], a crack growth path appears more tortuous when pearlite is arranged in bands instead of being random distributed. In our case, crack branching would results from the easy local plasticity at crack tip due to low hardness which generates a network of crack embryos. As a crack propagating in the ferrite matrix encounters a pearlite grains, it can cross easily especially



because of the large size of the ferrite and cementite lamellas. For secondary cracks pearlite act as obstacles, retard or arrest their propagation.

## 5. Conclusions

From this article, the following conclusions can be given:

- High temperature thermo-mechanical treatment changes the microstructure of the bainitic steel into a ferritic-pearlitic one.
- The hardening of the pearlitic steel is a lot more important than for bainitic steel ; the hardening of the ferritic-pearlitic steel is due mainly to ferrite grains whereas the relative stability of the bainitic steel is due to the competition between ferrite hardening and bainite softening.
- A load transfer exists in bainitic steel which causes ferrite to accommodate the plasticity more than bainite.
- However the nucleation and straight growth path of fatigue cracks in bainite is mainly due to the small amount of ferrite into the material.

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