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## **Fatigue intrusion-extrusion in a fully pearlitic steel**

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### **Abstract**

The aim of this paper is to identify the location of fatigue extrusions and intrusions, the latter being the precursor of short microcracks in a fully pearlitic steel. The material contains a huge amount of interfaces, as it consists of  $\alpha$ -ferrite bands separated by  $\text{Fe}_3\text{C}$  cementite lamellae.  $\text{Fe}_3\text{C}$  cementite remained inactive while cyclic plasticity occurred in  $\alpha$ -ferrite bands. Both intrusions and extrusions formed in the interior of the  $\alpha$ -ferrite bands. Unlike other materials, intrusion did not nucleate at the interface between  $\alpha$ -ferrite and  $\text{Fe}_3\text{C}$  cementite. The coherency of the interface is suspected to be responsible for this shift.

### **Keywords**

Deformation and fracture – Fatigue – Interfaces – Microstructure - slip markings

### **Introduction**

Fatigue failure is one of the major issues in many applications of materials. Fatigue life comprises a stage of crack initiation followed by crack propagation and sudden fracture. Crack initiation can occur either under or at the surface. There are several

possible sites for fatigue crack initiation such as grain boundaries, twin boundaries, inclusions, persistent slip bands [1]. The presence of interfaces therefore plays a crucial role on fatigue resistance. Cyclic loading above the yield stress results in the formation of fatigue slip markings (FSMs) at the external surface of the material. Classic examples of these are the intrusion-extrusion pairs. The evolution of intrusion-extrusion during the cyclic loading leads to the formation of short cracks. Their assessment can serve as quantitative indicators of accumulated fatigue damage [2]. The understanding of intrusion formation mechanism is, therefore of prime interest for the development of engineering materials resistant to fatigue. Hence, it is very important to identify, very accurately, the site of intrusion nucleation. The objective of the present paper is to investigate the formation of intrusion in a fully pearlitic steel, a material that contains a huge number of ferrite/cementite interfaces.

### **Experimental details**

The investigated material is an AISI 1080 carbon steel with the chemical composition given in Table 1.

Table 1: chemical composition of the studied pearlitic steel (wt%)

C	Mn	Si	Cr	Ni	P	S	Fe
0.776	0.587	0.235	0.020	0.020	0.014	0.009	Bal.

The material has been austenitized at 950°C then cooled and maintained at 640°C before final cooling at room temperature, resulting in a hardness Hv of  $312 \pm 9$ .

A low cycle fatigue strain controlled test has been performed on a cylindrical specimen. The external surface was mechanically polished beforehand to 0.25  $\mu\text{m}$ . The fatigue test was conducted at a total strain amplitude of  $\varepsilon_t=1.6\%$  under a mean strain of  $\varepsilon_m=0.9\%$ . After fatigue testing, the external surface was observed by SEM for observation of FSMs. TEM observations were performed on lamellae extracted directly under the FSMs by FIB. To protect the surface against the ion insertion, the area of

interest was covered by two different layers: a thin layer of carbon deposited by electron bombardment and a thick layer of platinum (about 1  $\mu\text{m}$ ) sputtered by ion bombardment. Firstly, FIB machined the craters from both sides of future lamella. Then the lamella was cut and transported on a copper grid and again thinned with a low ion energy beam (5-10 keV) in order to remove or minimize the damage caused by the previous steps. Imaging was performed on a FEI Titan Themis 300 analytical microscope working at an accelerating voltage of 300 kV.

## **Results and discussion**

The microstructure is fully pearlitic, consisting of a  $\alpha$ -ferrite matrix containing  $\text{Fe}_3\text{C}$  cementite of inter-lamellar spacing of  $170 \pm 20 \text{ nm}$  (Figure 1).

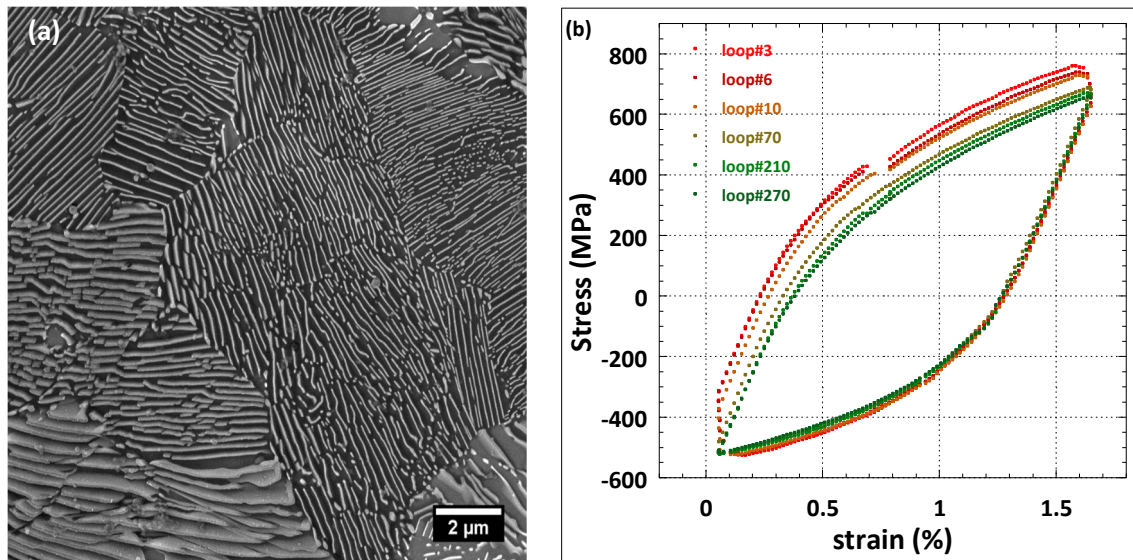


Figure 1: (a) optical micrograph of the fully pearlitic steel (white lamellae are  $\text{Fe}_3\text{C}$  and the dark background is  $\alpha$ -ferrite matrix and (b) hysteresis loops showing the cyclic softening

The stress response of the material under the imposed total strain of 1.6% consisted of a very slight softening (see Figure 1b). The ability to plastically deform was reflected by the presence of FSMs at the external surface (see Figure 2). These are very similar to

fatigue extrusions found in other grades of steels. Intrusions or short cracks can be seen inside the network of the slip markings, but cannot be easily distinguished by a simple observation of the external surface of the specimen. Cyclic plasticity was inhomogenously distributed since deformed areas were adjacent to non-deformed ones.

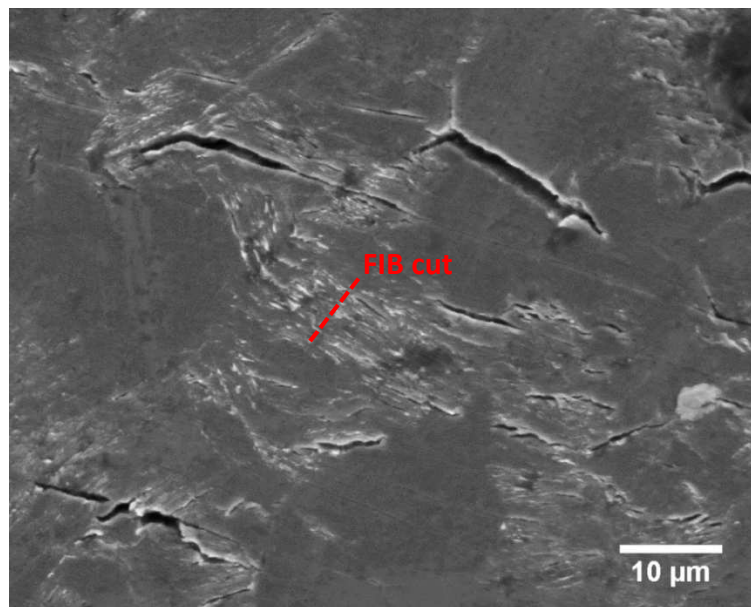


Figure 2: surface relief induced by cyclic deformation (SEM image) with location of FIB cut for further TEM observations

Because of the complexity of the pearlitic microstructure, the link between microstructure, short cracks, and intrusion-extrusion pairs is difficult to establish if the observation is performed from the external top face of the specimen. The TEM observations of the FIB lamellae allow overcoming this difficulty. It was observed that an extrusion and an intrusion were in fact formed inside the bands of  $\alpha$ -ferrite (see Figure 3). This intrusion evolved into short crack that propagated inside the  $\alpha$ -ferrite. The image clearly shows that the lip of the microcrack has a serrated shape with inclined dislocation slip traces.

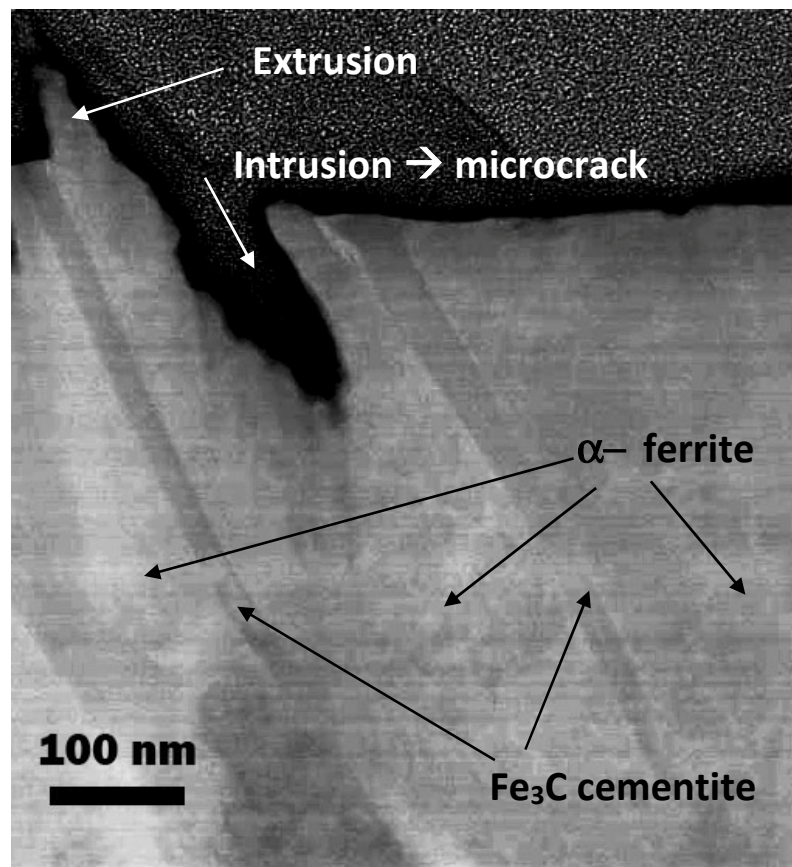


Figure 3: TEM image of the FIB lamella showing an intrusion involving into short crack and an extrusion in  $\alpha$ -ferrite

Several models of intrusion-extrusion formation have been developed in the literature e.g. [3 -5]. Among them, the model proposed by Polak and Man [4, 5] is certainly the most relevant and attractive one. They explained the sequence formation of extrusion and intrusion and the role of point defects, especially vacancies. In annealed copper or in solution heat treated austenitic stainless steel, cyclic straining results in a two-phase dislocation structure consisting of persistent slip bands (PSBs) embedded in a vein matrix. The authors emphasized the role of interface between the PSB and the elastically deformed matrix where an exchange by migration of vacancies and atoms provokes internal stress in the PSB which is relaxed by an emerging extrusion. In BCC steels cycled in the so-called high temperature regime, dislocations substructures

similar to that observed in wavy slip FCC metal can form. Thus, PSBs, walls and cells can form as well in equiaxed ferritic grains steels [6] as in elongated laths of martensitic [7] or of bainitic steels [6]. Recently the formation of extrusions and intrusions has been approved in a 12Cr martensitic steel [8] taking into account Polak and Man ideas [4, 5]. This material exhibits the classical hierarchic microstructure consisting of grains, packets, blocks, and laths. Intrusion-extrusion pairs were indeed observed and the location of intrusion was unambiguously found at the interface between two laths. It was concluded that the interface between martensitic laths in the 12 Cr martensitic steel behaved as the interface between the PSB and the matrix in other materials. The fully pearlitic steel investigated in this manuscript exhibits some similarities with this martensitic steel in terms of shape and size of scanned area by dislocations. As for thin martensite laths, dislocations were indeed observed in the narrow  $\alpha$ -ferrite bands; however, they were homogeneously distributed, not structured. No dislocation walls nor cells were observed, though plastic deformation activity was marked. Dislocation loops were observed to be emitted from the interface between  $\alpha$ -ferrite bands and  $\text{Fe}_3\text{C}$  cementite lamellae. The reason of the absence of structuration of dislocations produced by cyclic straining arises from the width of  $\alpha$ -ferrite bands. In comparison with large ferritic grains (10 to 50  $\mu\text{m}$ ) or with martensite lath (width between 0.4  $\mu\text{m}$  up to 1  $\mu\text{m}$  and length of about 10  $\mu\text{m}$ ), the width of  $\alpha$ -ferrite bands is much smaller (typically 0.14  $\mu\text{m}$ ). Consequently, dislocations cannot glide in the three space directions in agreement with their slip system and their Schmid factor but have an oriented and restricted displacement along the bands. Nevertheless, the gliding of these dislocations and their interaction can result in high production of vacancies as for other materials. All the conditions are present to form extrusions in  $\alpha$ -ferrite bands according to the above-mentioned mechanisms [4, 5], based on migration of vacancies toward an interface and atoms moving in the opposite direction. Regarding intrusions, they were also observed close to the extrusions and inside the  $\alpha$ -ferrite bands. Intrusion was nevertheless expected along  $\alpha$ -ferrite bands/ $\text{Fe}_3\text{C}$  cementite interfaces, which was not the case. This does not question the importance of the interface on intrusion nucleation, but suggests to focus on the

feature of the interface. First, in the investigated martensitic steel, the interface joins materials of same nature as well in terms of chemical composition, mechanical response, and average crystallographic orientation. Indeed, the interface is a low angle boundary. In the pearlitic steel, the situation is exactly opposite: difference in chemical composition, crystallographic structure, and mechanical response (cementite is much harder than ferrite). In addition, the interface is likely to be coherent in regards with the literature [9]. Finally, the situation looks like a “composite” with a quasi non plastically deformable phase ( $\text{Fe}_3\text{C}$  cementite) beside a deformable one ( $\alpha$ -ferrite). It turns out that facing with a coherent interface could modify the location of vacancies sinks and atom sources. It seems that there exists a band separating the very interface between  $\alpha$ -ferrite and  $\text{Fe}_3\text{C}$  cementite and the  $\alpha$ -ferrite bands where fatigue plasticity occurs. This band acts as a shield forcing vacancies produced in the active  $\alpha$ -ferrite bands to diffuse and accumulate ahead of the interface between  $\alpha$ -ferrite and  $\text{Fe}_3\text{C}$  cementite. It is also interesting to note that despite  $\text{Fe}_3\text{C}$  cementite being a hard phase, it did not break nor initiate crack as it can be observed in a  $\text{TiB}_2$ -reinforced steel matrix composite [10].

Additional investigations are currently undertaken to have a more precise description of this shield band.

## **Conclusions**

Under cyclic loading, the fully pearlitic steel exhibited well-developed slip markings. Extrusions were easily observable by SEM at the external surface of the specimen. TEM observations on FIB lamellae revealed that both intrusion and extrusion formed in the  $\alpha$  ferrite phase. Intrusion initiation site was slightly away from the interface between  $\alpha$  ferrite phase and  $\text{Fe}_3\text{C}$  cementite interface.

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