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Measurement of small cracks initiating from inclusions, Focused Ion Beam notches and drilled holes

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ABSTRACT

The focus of this study is small crack initiation, growth and arrest from small defects in quenched and tempered steels. To study the initiation and growth threshold of microcracks from small defects a specialised test method was developed. Round bars were axially loaded at $R = -1$ and fatigue initiators were manufactured by drilling (∅ 50 μm) and Focused Ion Beam (FIB) milling (a = 10–50 μm). The specimens were fatigued at the range of the smooth specimen fatigue limit. The initiated microcracks were monitored to observe crack initiation; growth and arrest. The high correlation of the FIB small crack growth results with inclusion test results indicates that using a FIB notch to initiate small cracks is a good way to test small crack growth in high strength steels.

1. Introduction

In fatigue of high strength steels the early growth mechanisms, paths and rates of the microstructurally small cracks are not well known. Small cracks tend to initiate from stress concentrations or the largest non-metallic inclusions within the stressed volume of modern high strength steels. Fatigue crack nucleation and growth from surface or subsurface non-metallic inclusions in steels is of importance in many industries. It has been the focus of many studies [1–6] and a book by Murakami [7] However, many questions still remain relating to the initiation and growth of microstructurally small cracks in high strength steels. Recent studies have used FIB milling to create small notches into different materials to study the growth of small cracks emanating from notches[8–18]. In this study we apply this method to smooth specimens and investigate the relevance of using FIB notches as small crack initiators. They are applied to measure small crack growth thresholds and are compared to small defects such as non-metallic inclusions in the steel.

2. Experimental method

In this study axial fatigue tests were performed for quenched and tempered wrought steel 34CrNiMo6 with 1065 MPa and 1180 MPa yield and tensile strengths respectively. The martensitic microstructure of the steel normal to the forging direction is shown in Fig. 1. The test bars were machined from the centreline of Ø35 mm wrought bars. The test bar dimensions are shown in Fig. 2.

Fatigue tests for all specimens were performed using a Rumul pulsator in laboratory conditions at room temperature with resonant frequencies that varied from 100 to 200 Hz. For the ultra long fatigue tests resonant frequencies close to 200 Hz were used. The lower frequency (⩾104 Hz) were applied for the testing of notched specimen. Small fatigue initiators were manufactured by drilling (∅50 μm) and FIB milling (a = 10–50 μm) into the test bars as shown in Fig. 2. The size of the FIB notches and drilled holes were chosen so that they could be compared to the natural occurring defects in the steel typically ranging from 10 to 40 μm. The maximum notch size of 50 μm was chosen to exceed the largest natural defect that we observed in over 50 test specimen. The FIB milling was done with a FEI Helios Nanolab dual focused beam system. The FIB was set to a voltage of 30 kV and a milling current of 21 nA was used. The pattern was defined in a diamond shape seen in Fig. 3e and f and Fig. 4 with a notch height typically of 5–10 μm.

The growth of small cracks was also studied using one special multi-notch test bar into which was milled altogether 20 small FIB notches in five rows and each row having four notches of 40, 30, 20, and 10 μm in length. An overview of the layout of the notches is given in Fig. 2 and a SEM picture of one of these notches is shown in Fig. 4. The distance between notches was greater than 390 μm and the test was stopped before the cracks grew to a size larger enough to interact in any major way. A mixed distribution round the specimen was applied to avoid systematic errors e.g. due to bending. Afterwards, the results proved that no bending

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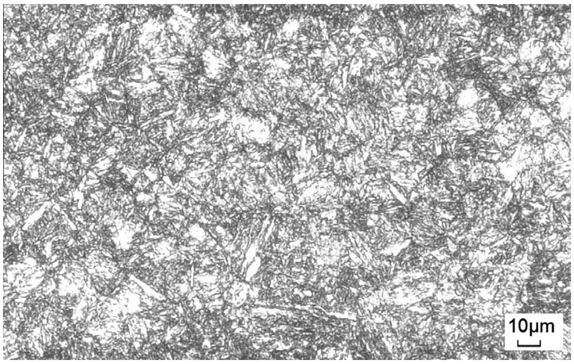

Fig. 1. Microstructure of the steel studied taken normal to the forging direction.

occurs or interaction occurs. The notched specimen was fatigued for 10,000 or 20,000 cycles at a time and then carefully studied by SEM to observe crack initiation and growth. The test was conducted at $R = -1$ and at a constant amplitude of 590 MPa. The stress range applied in the tests was close to the fatigue limit of the steel which was determined from SN test data of 30 specimen to be around 570 MPa. Therefore the crack growth behaviour which was observed corresponds to crack growth rates near the threshold for crack growth initiation and arrest.

The small holes and notches were monitored optically during the tests to detect small crack initiation and growth. Once the test bar failed or the test was complete it was examined with SEM, laser and optical microscopes. Sometimes the test bars failed from some other location such as an inclusion. Then the nucleation site was studied with a SEM and the type, size, and shape of the nucleation site was recorded. An overview of the test method is shown in Fig. 3.

3. Calculation of stress intensity factors

Two different types of measurements are reported in this paper. One type of measurements was done from the fracture surfaces (Figs. 7 and 8). These results are analysed and presented using the $\sqrt{\text{area}}$ of the small crack and/or initiating defect on the cross-section normal to the stress. The threshold for crack growth or initiation for the different sizes of notches, holes and inclusions were calculated by using the following equation [7]:

$$\Delta K_{th} = 0.65 \cdot \Delta\sigma \cdot \sqrt{\pi\sqrt{\text{area}}} \quad (1)$$

The other type of measurement was observed during testing, either in situ or during interrupted loading (Figs. 5 and 6). These results are reported as crack lengths on the specimen surface. The length of the surface crack was used to calculate the stress intensity factor range for the FIB notch test bar by using the following equation:

$$\Delta K_{th} = 0.65 \cdot \Delta\sigma \cdot \sqrt{\pi a} \quad (2)$$

In Eq. (2) the variable 'a' is from the observed crack length of '$2a$'. Any crack solution becomes arbitrary, because continuum fracture mechanics are no longer valid in the studied dimensions. The same shape factor (0.65) was applied in both equations. This shape factor value is arbitrarily selected within a wide range of values generally used in literature [19]. Its value just affects the horizontal axis scale position in Fig. 6. In geometrical terms this would equal to an aspect ratio of $\frac{a}{c} = \pi/2$, where c is crack depth of a semi-elliptical crack.

4. Results

4.1. Multi-notch specimen (FIB)

The test bar with 20 small FIB notches gave a large and interesting set of results, because crack growth was observed in almost all notches irrespective of their different sizes. The observed crack growth is shown in Fig. 5.

The data in Fig. 5 can be used to calculate the crack growth rate versus the stress intensity factor range. This was done by averaging the crack growth rate as well as the stress intensity factor range over each observation interval. These results are illustrated in Fig. 2 where they are separated into original notch sizes. To illustrate the variation of growth rate along the cycle count, another grouping of the same data is also shown in Fig. 6.

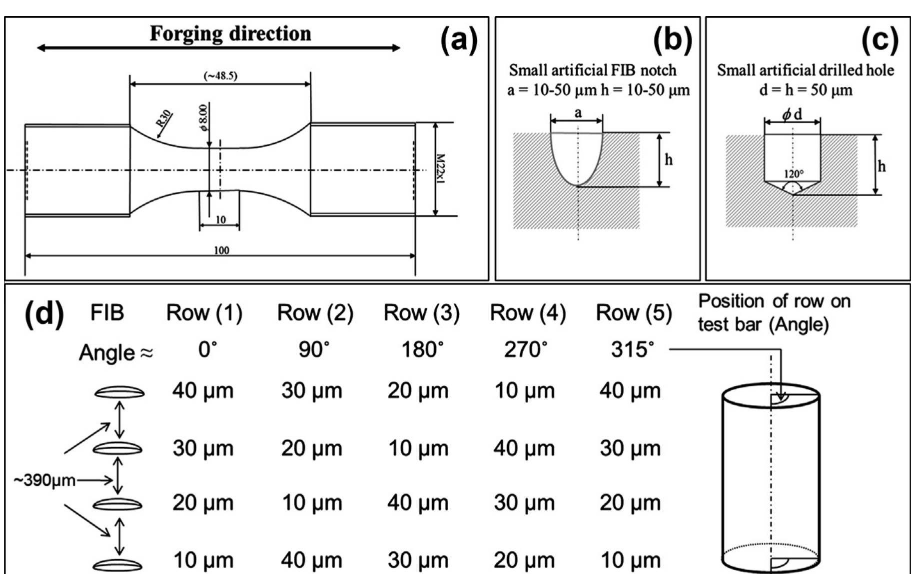

Fig. 2. The dimensions of the test bar are shown in (a) with the shape, size, and types of holes made in the side of the test bars shown in (b and c). The postions and placement of the FIB notches with respect to the test bar are shown in part (d) for the multi-notch test bar that was tested with 20 FIB notches.