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Crack arrest testing at the micro-scale

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ABSTRACT

Crack arrest testing of micro-sized cantilever beams ($\approx 8 \times 4 \times 6\,\mu m$, length, width and height, respectively) was conducted in order to evaluate the suitability of a new method to quantify local crack arrest properties. Chevron notched cantilevers were milled to match the (1 0 0)[0 $\bar{1}$ 1] crack system in α-iron, where earlier attempts to obtain brittle or rapidly propagating fracture proved difficult. Brittle crack initiation and propagation was achieved by means of the deposition of a layer of SiO_X on the surface, acting as a brittle starter. All tests were performed at −75 °C, using an in-house designed cooling system. The cracks arrested after propagation into the iron cantilever. A finite element model was developed to determine the appropriate dimensionless shape factor and provide a rigorous computer analysis of these complexly shaped cantilevers. K_{QC} and K_{Qa}, at initiation and arrest respectively, were determined and evaluated. The cantilevers were later displaced further at 40 K to allow evaluation of crack jump lengths and to obtain a more complete analysis of the fracture surfaces. The average fracture toughness was determined to be $3.89 \pm 1.00\,MPa\sqrt{m}$, and the average arrest toughness to be $2.6 \pm 0.86\,MPa\sqrt{m}$. The finite element model highlights the effect of small variations in geometry which was larger than anticipated and strongly affects the shape factor, up to a 25% difference in f(a/W). As small variations in geometry are inevitable when milling with FIB, the need for individual models tailored to every cantilever is discussed.

1. Introduction

Traditionally, fracture mechanics research has been conducted at a macroscopic scale with focus on statistical treatment of fracture, ductile to brittle transition, and determination of the critical parameters inducing fracture through a local approach [1–6]. The quantification of the fracture parameter values, particularly in relation to the different steps in the fracture process is considered crucial in order to understand the deformation mechanisms at a microstructural level. One of the most recognized local approaches to fracture is the so-called Multiple Barrier model, introduced by Lambert-Perlade et al. [7] which defines the whole fracture phenomena as a stepwise process where several individual barriers are required to be overcome for final fracture to happen. The work presented in this paper aims to demonstrate the suitability of the test methodology in establishing crack arrest, increase the understanding of the crack arrest phenomena in metals, and eventually provide quantitative values for the Multiple Barrier model. Once the fracture toughness and the governing failure mechanism for individual microstructural aspects is quantified and understood from a local approach to fracture point of view, the dynamic between micro and macro can be approached.

The development of testing methods towards the definition of crack arrest properties of individual grains, grain boundaries and

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other important microstructural aspects, such as martensite-austenite (MA) constituents, is imperative to gain complete insight into the governing factors of local fracture. As both offshore and onshore operations are moving into the Arctic regions, where low temperatures drastically affect the fracture toughness of most steels, a deeper understanding of the crack arrest phenomena becomes increasingly important. Determining the micro-arrest toughness is a necessary step towards this direction, allowing a knowledge-based application of a multiple barrier model for which the quantification of the material properties is therefore crucial. Standards dedicated to testing and determination of valid fracture mechanics parameters are based on relatively large samples (macro-scale and mm-sized) rendering determination of micro-scale fracture toughness values impossible or inappropriate. Hence, small-scale testing methods obtaining non-valid fracture parameters are used to quantify crack arrest properties of individual microstructural aspects. For instance, Argon and Qiao showed the importance of grain boundary orientation, and grain boundary tilt and twist, on crack arrest in Fe-3%Si [8–10], while Hribernik tested the arrest toughness of iron grains [11]. However, since the tests carried out by these two groups were performed on millimeter-sized samples, the versatility and therefore the information possible to gain through these testing methods is reduced. Reducing the size of the samples to the micrometer regime enables specific probing of micrometer-sized microstructural aspects such as MA constituents, steels with small grain sizes, and heat affected zones from welding [12].

The amount of publications focused on fracture experiments on micro-sized cantilevers is somewhat limited, but steadily increasing in the last decade [13–23]. As the sample size is reduced to micro- and nanoscale, the continuum mechanics assumption are no longer satisfied pushing the fracture tests beyond the validity limits defined in the ASTM standards and therefore obscuring failure predictions [24]. This is especially true and relevant for ductile materials, i.e. materials where the plastic zone size in front of the crack is comparable or larger than the specimen cross-section. However, linear-elastic fracture mechanics have been successfully applied to brittle materials, yielding comparable values for large- and small-scale samples [15–17,19]. This paper presents crack arrest testing at the micro-scale level, and is, to the author's knowledge, the first of its kind. In order to obtain a running brittle crack, a layer of brittle material was deposited on the iron cantilever beam. In this way, a brittle crack is propagating into a more ductile material, so that the resulting arrest of the crack would enable the quantification of the micro-scale arrest toughness.

2. Experiments

The pure iron cantilever notch orientation used in this experiment was $(1\,0\,0)[0\,\bar{1}\,1]$, where $(1\,0\,0)$ and $[0\,\bar{1}\,1]$ refer to the crack plane and crack front direction, respectively. This particular orientation was chosen based on results from atomistic modeling [25], that revealed brittle behavior over dislocation emission for the $(1\,0\,0)[0\,\bar{1}\,1]$ crack system. The cantilevers were designed to achieve an initial brittle fracture; a brittle layer was deposited at the surface and within the milled Chevron notch, side grooves were added to increase the cantilever's compliance and the test was run in load-control, see Fig. 1. A Chevron notch has a triangular ligament, so that the width of the section increases as the crack grows. This geometry, initially developed to obtain valid fracture parameters for brittle materials as ceramics, is in fact well suited for crack arrest experiments as it promotes stable crack growth: a crack propagating in an unstable manner will tend to arrest as the driving force decreases with crack growth in an increasing material front.

The specimens were fabricated from a sample of 99.99% pure iron, with chemical composition given in Table 1, and heat treated to enlarge the grain size. The pure iron was polished using 1 µm grit diamond, and electro polished at 35 V for 20 s using an A2 electrolyte. Electron BackScatter Diffraction (EBSD) analysis was conducted to individuate grains with orientation $(0\,1\,1)$. The crack system $(1\,0\,0)[0\,\bar{1}\,1]$, was obtained by pinpointing the $(0\,1\,1)$ orientation, and rotating the sample according to the unit cell orientation within the highlighted grains. By aligning the $(1\,0\,0)$ direction of orientation $(0\,1\,1)$ perpendicularly to the produced cantilevers, the desired aforementioned crack orientation was obtained.

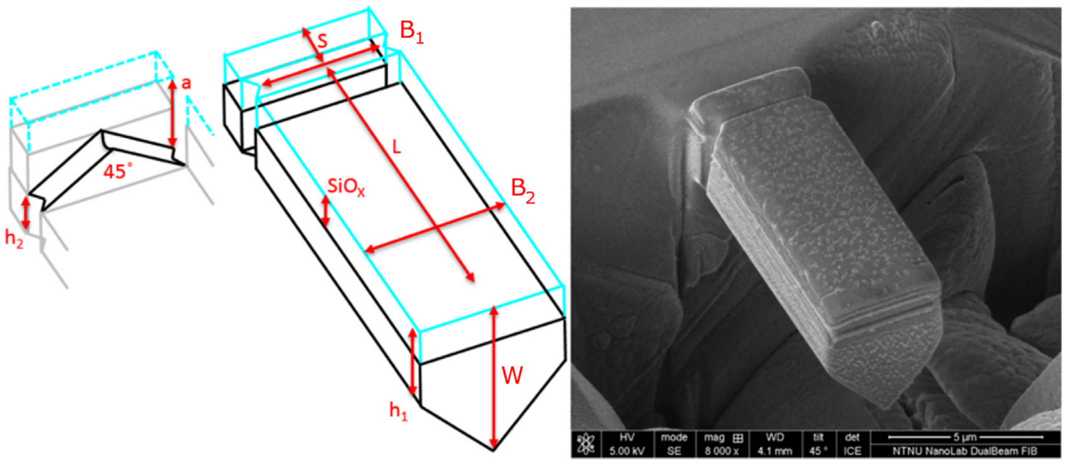

Fig. 1. Schematic illustration of a cantilever beam with side grooves, Chevron notch (left). and deposition layer. Red arrows indicate relevant dimensions. Micrograph of a finished cantilever with deposition layer is presented to the right. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)