Supporting Information

Quantitative in situ SEM High Cycle Fatigue: the Critical Role of Oxygen on Nanoscale-Void-Controlled Nucleation and Propagation of Small Cracks in Ni Microbeams

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Supporting Materials and Methods

Section A: In situ SEM Experimental Technique

The fatigue tests consist of the in-plane vibration of a microresonator shown in Figure 1(a). The beam and fan shaped mass assembly is electrically grounded and the input of a high sinusoidal voltage to one of the neighboring comb structures promotes the vibration of the microresonator via electrostatic forces. The microbeam attached to the microresonator experiences fully reversed bending fatigue when it is actuated at its resonance frequency, f_0 (~8 kHz). The other neighboring set of combs is used to sense the second harmonic capacitive current across the comb fingers (through the input of a bias DC voltage) and is converted to a voltage signal with a custom made circuit[1] that is outside the SEM. The output voltage signal is proportional to the amplitude of angular displacement of the microresonator, θ_0.[2] The device is periodically swept over a small range of frequencies around the expected f_0 in order to track its changes (see example in Figure S1(a)). The resonator is then continuously driven at the previously found f_0, which decreases with cycling as the fatigue damage on the beam continues to develop. The in situ SEM fatigue tests are performed in a FEI Nova Nanolab 200 FIB/SEM, using electrical feedthroughs to apply the electrostatic force onto the actuation comb drive, and sense the induced current from the sensing comb.

Since the sensing capabilities of the structure do not allow for an absolute value of the angular displacement amplitude θ_0, an optical calibration is performed at the beginning of each fatigue test. The optical calibration consists of measuring the distance between the edge of a comb's finger at rest (when no load is applied) and the edge of a comb's finger vibrating at the resonance frequency (maximum θ_0), as shown in Figure S1(b) and (c). The input of a high voltage in the SEM causes the electron image to become blurry, adding some uncertainty to the θ_0 measurement. The uncertainty is found by measuring the blur of the stationary combs in the SEM images, shown by the red arrow in Figure S1(c), and typical values are around 1 mrad. Once the initial θ_0 of the microbeam is found, the stress amplitude (σ_a) is calculated using the relationship that was obtained by finite element modeling[3] of the entire microresonator, using the measured mechanical properties of the electroplated Ni[1] (see Figure S2(a)). All the stress amplitudes in this study refer to the initial stress amplitude value at the edge of the microbeam. The uncertainty in σ_a is ~20 MPa, based on the uncertainty in the angle of rotation at resonance (see Figure S2(a)). Figures S2(b) and (c) provide the overall trend in the measured θ_0 and corresponding σ_a, respectively, as a function of the amplitude of input voltage, V_{in}, for all tests performed in air and vacuo. The larger values in vacuo result from a slight increase in the microresonator's quality factor in the absence of air damping (the quality factor is dominated by dislocation damping for that range of input voltage amplitude[1]).

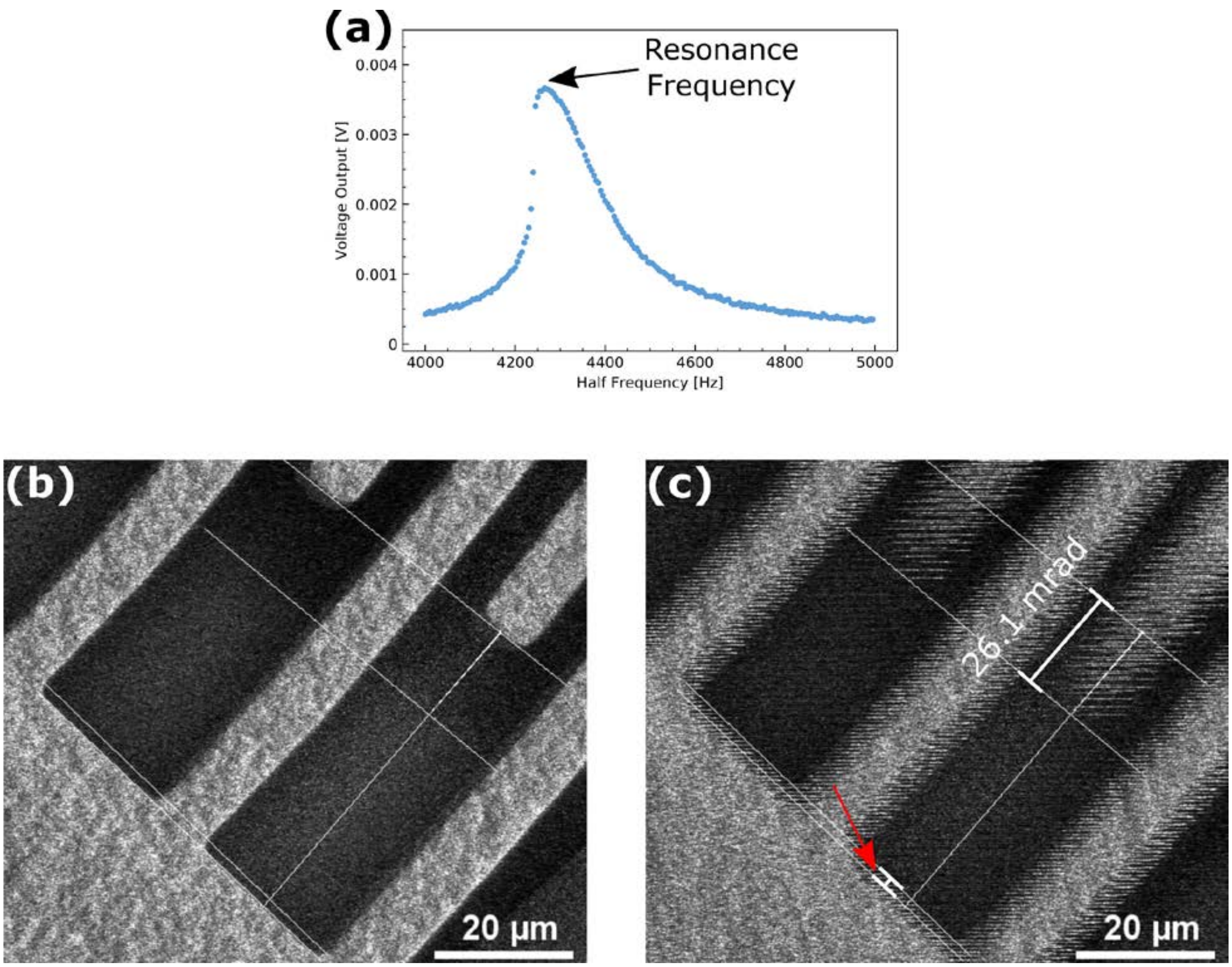

Figure S1- (a) Example of a sweep for a fatigue test in air at an input amplitude voltage of 200V. SEM images of the combs for the optical calibration when the device is (a) at rest and (b) vibrating at its resonance frequency.

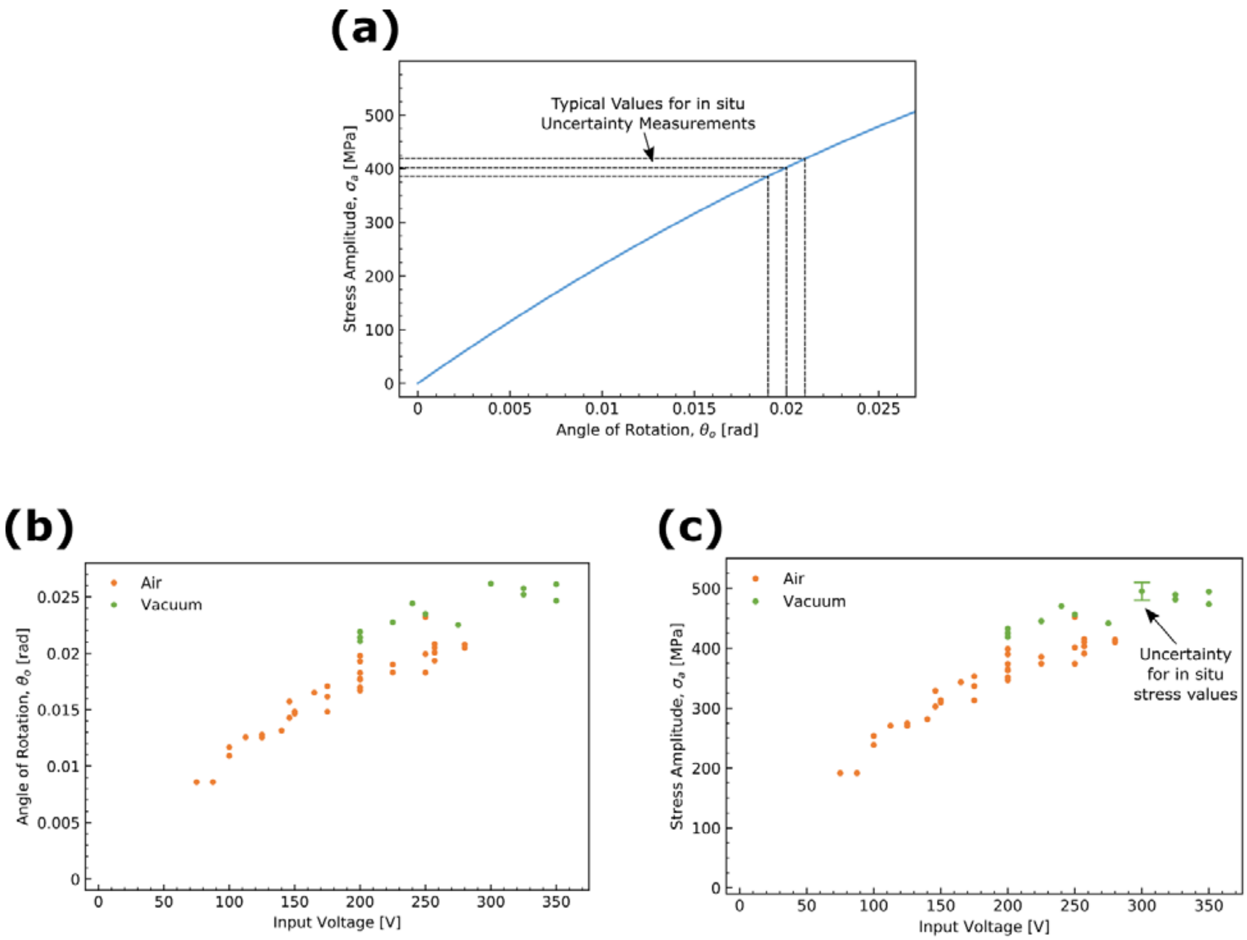

Figure S2- (a) Finite element modeling relationship between angle of rotation and stress amplitude. [3] Relationship between input amplitude voltage and (b) the angle of rotation and (c) the stress amplitude of fatigue tests done in air and in vacuo.

Section B: Environmental Effects on Crack Growth Rates for a Single Specimen

Figure S3(a) shows the f_0 evolution plot of a fatigue test consisting of cycling a specimen at σ_a = 390 MPa in air for 4.8×10^6 cycles, followed by cycling in vacuo until 7.4×10^7 cycles at σ_a = 360 MPa. Two cracks, one on each side of the microbeam, developed in air. Between stops (b) and (c) (see Figure S3(a)), the crack grows (compare Figures S3(b) and (c)), based on the surface crack length, at an average rate of 2.0×10^{-12} m/cycle. The crack did not extend in vacuo at σ_a = 360 MPa between stops (c) and (d), but extended between (d) and (f) (See corresponding SEM images in Figure S3) at a rate of 3.7×10^{-14} m/cycle. See also Animation 2 in the Supporting Information.

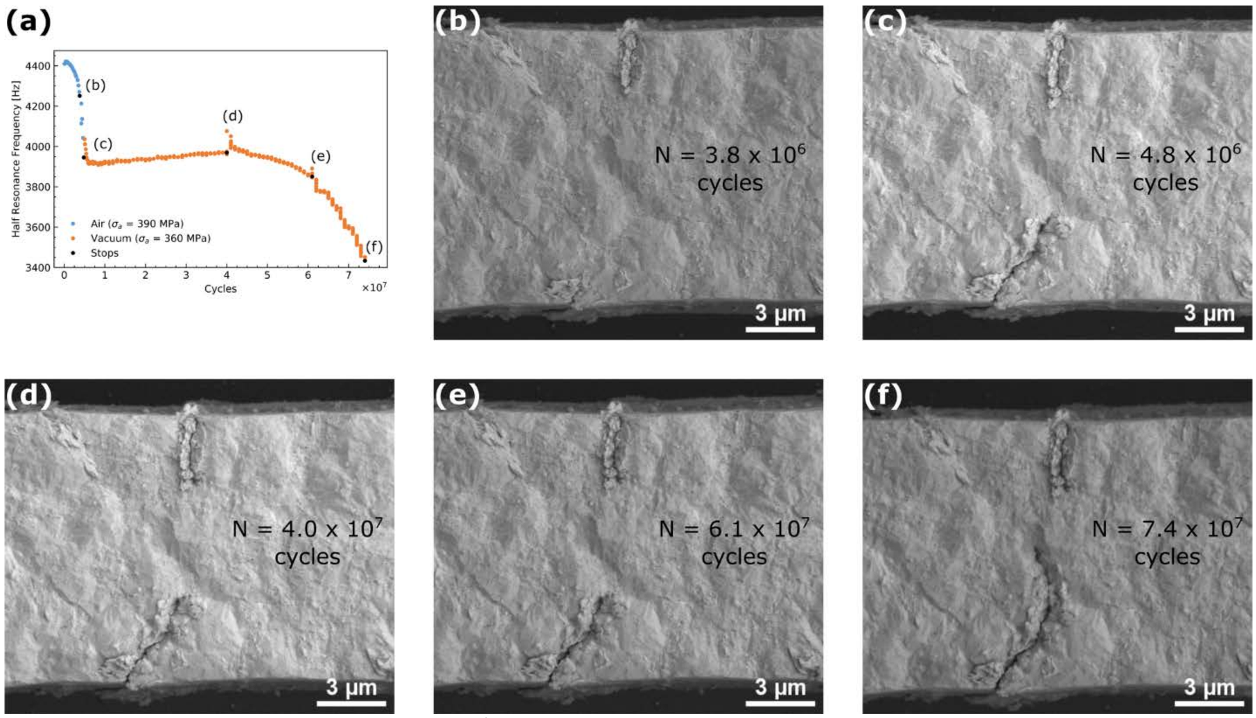

Figure S3- (a) Frequency evolution ($\frac{1}{2} f_o$) during an ex situ test in air performed at σ_a = 390 MPa followed by SEM testing at σ_a =360 MPa. (b) to (e) Top-down SEM images of the microbeam's top surface, highlighting the propagation of the crack towards the neutral axis. Each image was taken upon stops as shown in black in (a).

Section C: Crack growth rates as a function of crack size

See Figure 1(d) for the corresponding graph showing the same crack growth rates as a function of stress amplitude.

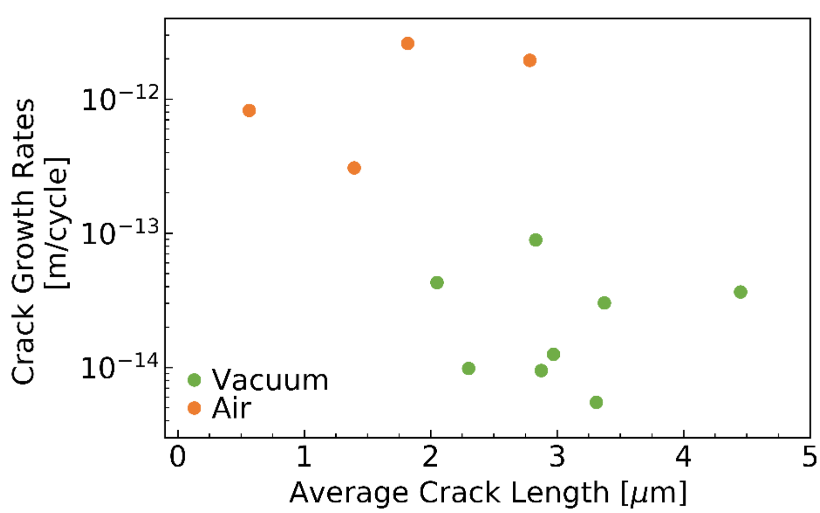

Figure S4- Crack growth rates as a function of average crack size

Section D: FIB Serial Sectioning and Imaging for Fatigue Specimen in Air

Figure S5(a) shows the results of a fatigue test in air at σ_a =390 MPa. Three sets of serial FIB sectioning and imaging were performed at three different locations on this specimen, shown in Figures S5(b)-(d). The corresponding animations showing the serial imaging are in the Supporting Information (Animations 3-5).

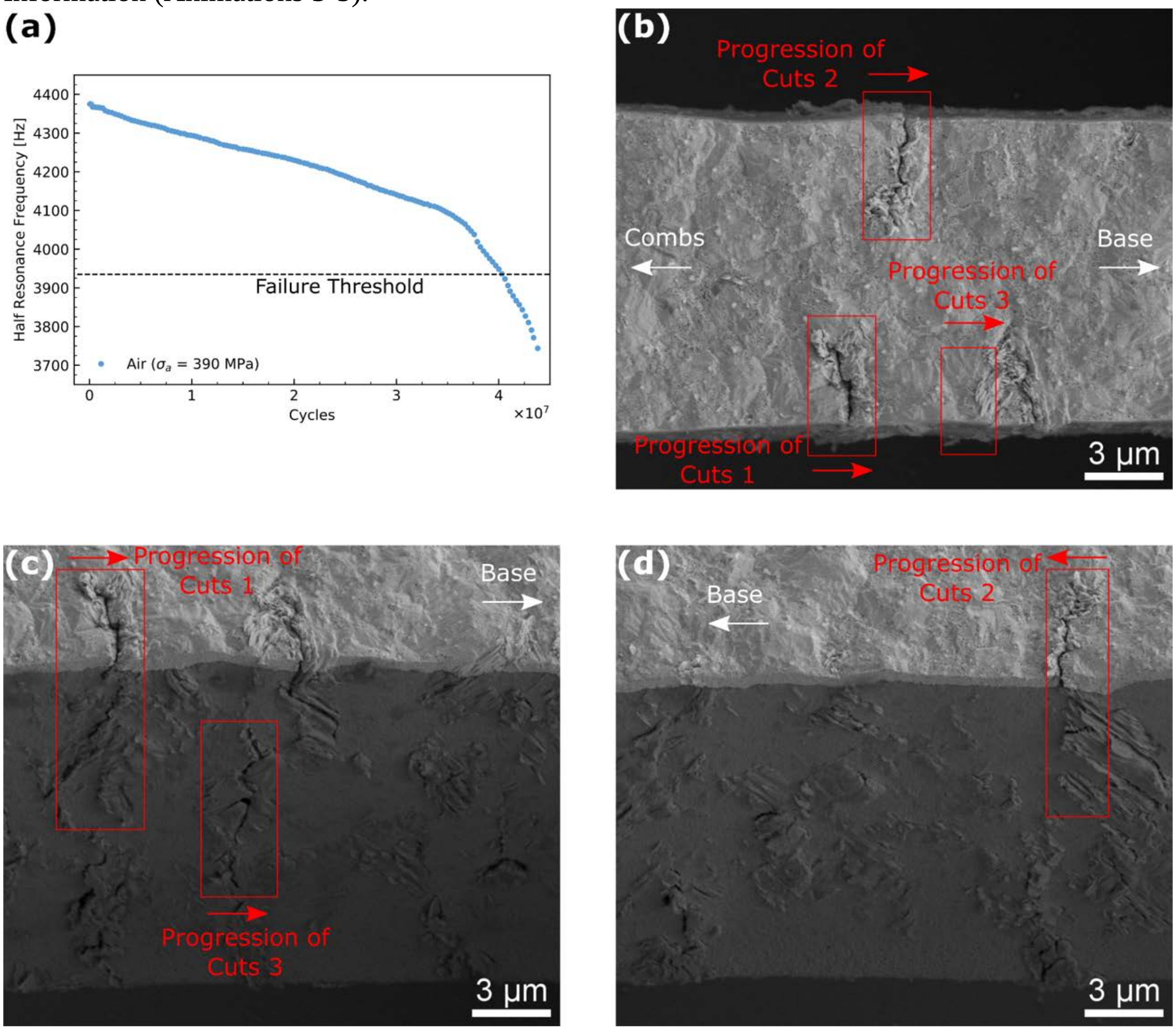

Figure S5- (a) Frequency evolution of a fatigue test in air. Location of the 3 FIB serial sectioning from (a) top view, (b) inclined view of one sidewall and (c) inclined view of the other sidewall

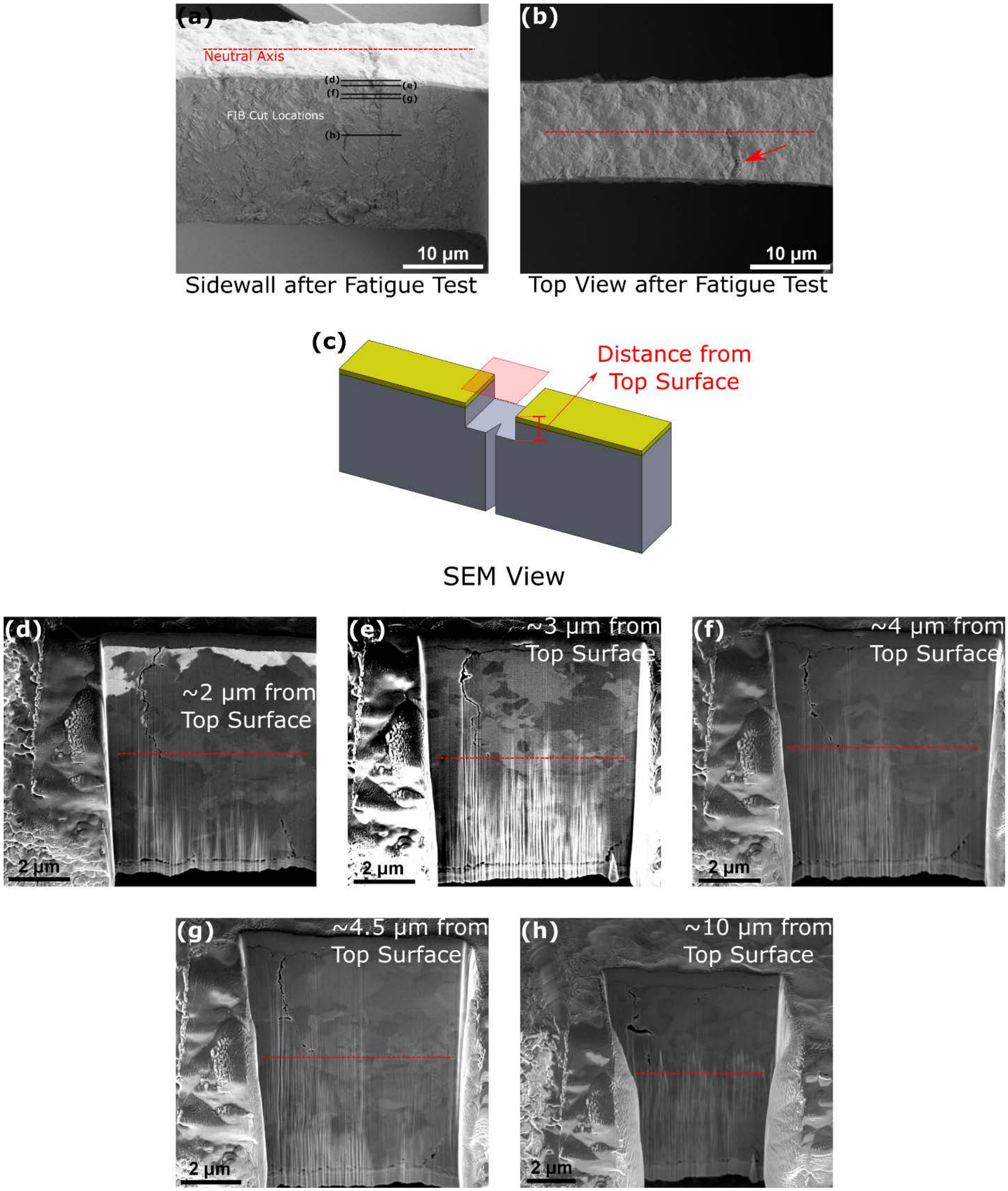

Figure S6- (a) Location of horizontal FIB cuts along sidewall. (b) Top view SEM image of microbeam after fatigue test. The red arrow indicates the location of the fatigue cracks. The red dotted line is the neutral axis. (c) Schematic showing the definition of distance from top surface in the remaining images. (d)-(h) SEM images showing overall shape of the fatigue crack at the various locations. The red dotted lines represent the neutral axis.

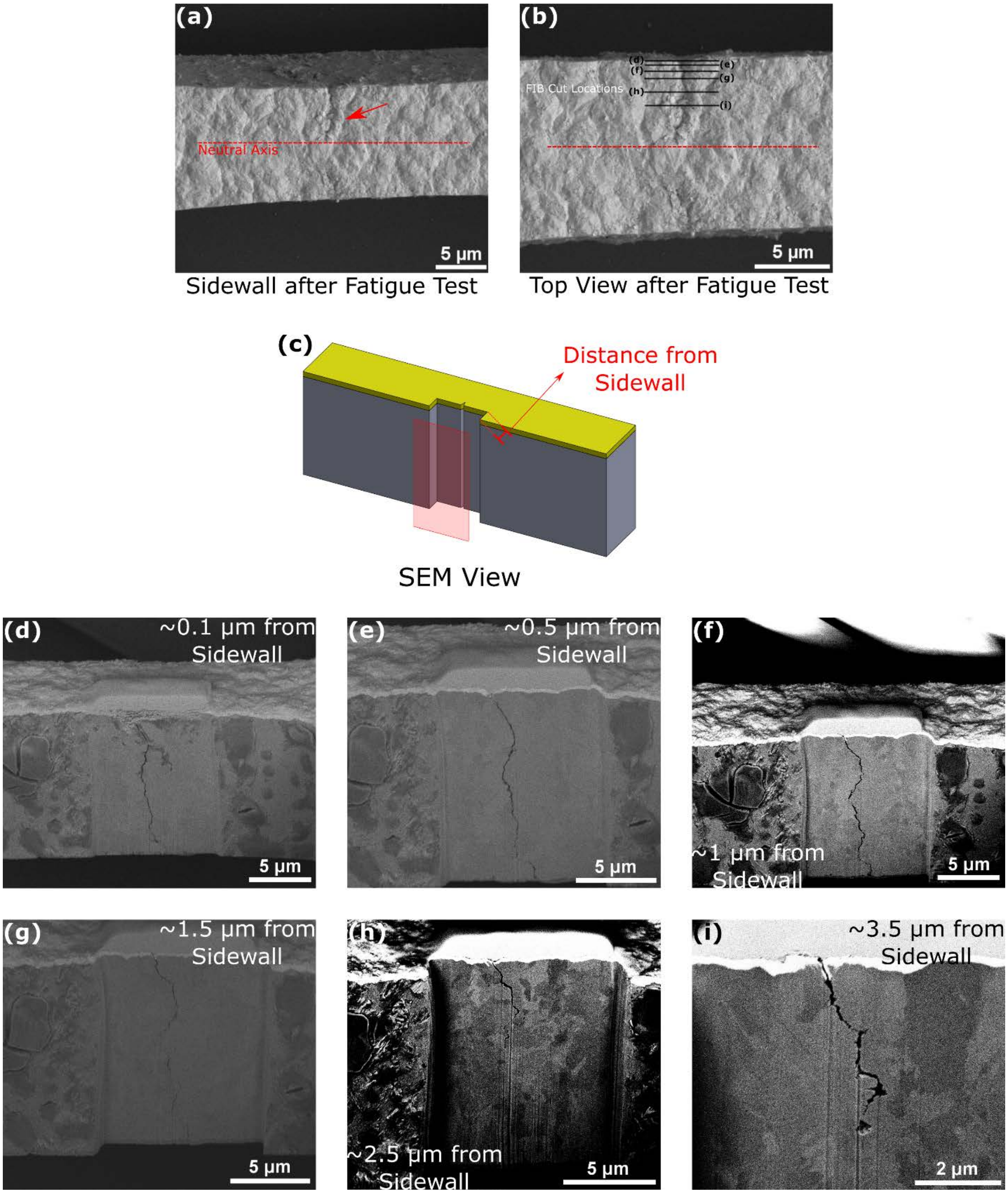

Figure S7- (a) Inclined SEM image after fatigue test; red arrow indicates fatigue crack location (b) Top view SEM image of microbeam after fatigue test with location of vertical FIB cuts. The red dotted line is the neutral axis. (c) Schematic showing the definition of distance from sidewall in the remaining images. (d)-(i) SEM images showing overall shape of the fatigue crack at the various locations. The crack is through the entire microbeam's thickness in (d)-(g), i.e., up to ~2 µm from sidewall. In (h) and (i), the crack is present in the top half of the microbeam's thickness.

References

1. Baumert, E. K.; Pierron, O. N. *Microelectromechanical Systems, Journal of* **2013,** 22, (1), 16-25.
2. Pierron, O. N.; Abnet, C. C.; Muhlstein, C. L. *Sensors and Actuators, A: Physical* **2006,** 128, (1), 140-150.
3. Sadeghi-Tohidi, F.; Pierron, O. N. *Extreme Mechanics Letters* **2016,** 9, 97-107.