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Delayed Fracture due to Time-Dependent Damage in PDMS

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What do we mean by "delayed fracture?"

A structure with or without an obvious crack or stress concentration may sustain a fixed load or fixed displacement for some time followed by sudden failure.

This is not accounted for by basic failure criteria such as

- Failure occurs in un-notched material when max tensile stress > tensile strength,
 - i.e. $\sigma_{max} > \sigma_u$
- Or, in a sample with a pre-existing crack, failure occurs when applied stress intensity factor exceeds fracture toughness, i.e.
 - $K_I > K_{Ic}$
- Our working hypothesis is that delayed fracture in our experiments is a result of time dependent bond failure.



Under constant load – creep rupture is a type of delayed fracture

- Crack may grow very slowly e.g. in metals at high temperature
- Or may be stable for some time and then suddenly fail
 - Example: PVA Hydrogel



Poly (vinyl alcohol) (PVA) dual-crosslink hydrogel

- Cross-linked by permanent (covalent) bonds and transient (physical) bonds.
- Breaking and healing of physical bonds results in highly viscoelastic behavior

Creep rupture in PVA Hydrogel – experimental setup

PID control to hold force at setpoint



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Mincong Liu, Jingyi Guo, Chung-Yuen Hui, Alan Zehnder, *Crack tip stress based kinetic fracture model of a PVA dual-crosslink hydrogel,* **Extreme Mechanics Letters,** Volume 29, 2019, 100457,

Creep rupture in PVA Hydrogel – experimental data

Stretch under constant nominal stress increases over time



Time to fracture is exponential with stress



A lot of scatter. Reducing stress by 2X increases time to failure by about 500 X

Delayed fracture of PDMS Under Fixed Stretch and Constant Stretch Rate

PDMS cross linked at 10:1 ratio selected to <u>minimize viscoelastic deformation</u> and focus on time dependent bond breaking.

Polydimethylsiloxane (PDMS)





Torsional rheometry data from 10:1 PDMS shows very low loss modulus – indicating low viscoelasticity



Delayed fracture experiments on PDMS



Double edge crack (DEC) sample with blunt crack tip





Result for blunt crack under step stretch followed by hold



Deformation is time-dependent and non-recoverable

Onset of unstable fracture in blunt crack samples



Sample failed after 7 hours holding at nominal stretch of 1.6

Images acquired continually at 200 fps. We are looking just at the last few frames prior to unstable failure

See also: H. M. van der Kooij, S. Dussi, G. T. van de Kerkhof, R. A. Frijns, J. van der Gucht, J. Sprakel, *Laser speckle strain imaging reveals the origin of delayed fracture in a soft solid*, **Science Advances 4** (5) (2018) eaar1926

Crack propagation from sharp crack under step stretch and hold



Crack speed from experiments with constant stretch rate combined with data from step stretch experiment



For each stretch ratio we can compute energy release rate

Constitutive model fit from uniaxial test data using Yeoh model

$$W(I_1) = \sum_{k=1}^{3} c_k (I_1 - 3)^k$$
$$I_1 = \lambda^2 + \frac{2}{\lambda}$$

For pure shear sample with long enough crack $(a/H \ge 1)$:

 $G_{PS} = W(I_1)H$ $I_1 = \lambda^2 + \frac{1}{\lambda^2} + 1$



Putting step and constant stretch rate data together with G



Kinetic (Eyring type) failure model for crack growth

Time-dependent chain breaking function

$$\frac{db}{dt} = 0, \qquad I_1 \le I_c$$

$$\frac{db}{dt} = -\frac{n_m}{\tau} b \exp\left(\frac{L_a f}{k_B T}\right), I_1 > I_c$$

$$\frac{db}{dt} = -\frac{n_m}{\tau} b \exp\left(\frac{L_a}{l_k} \sqrt{\frac{I_1(B)}{3n}} \left[\frac{3 - \frac{I_1(B)}{3n}}{1 - \frac{I_1(B)}{3n}}\right]\right)$$



Crack speed calculation

From FEM simulation, for each nominal stretch, λ , we have $I_1(x)$ and hence force on chains, f is known f = f(x)

For steady state crack growth

$$\frac{db}{dt} = -v \frac{db}{dx} = -\frac{n_m}{\tau} b \exp\left(\frac{L_a f}{k_B T}\right), v = \text{crack speed}$$

Set $\frac{db}{dt} = 0$ for $I_1 < I_c$ at distance x = L ahead of crack tip, b = 1 at x = L.

Failure condition is $b = b_c$ at $x = x_c$

Integrate the above and solve for v:

$$v = \frac{n_m}{\tau \ln(1/b_c)} \int_{x_c}^{L} \exp(\frac{L_a f(x)}{k_B T}) dx$$



Model Parameter Identification

Crack speed result from prior slide (writing out *f*, force acting on chain)

$$v = \frac{n_m}{\tau \ln\left(\frac{1}{b_c}\right)} \int_{x_c}^{L} \exp\left(\frac{L_a}{l_k} \sqrt{\frac{I_1(B)}{3n}} \left[\frac{3 - \frac{I_1(B)}{3n}}{1 - \frac{I_1(B)}{3n}}\right]\right) dx$$

Five independent parameters in the model: $\{\tau \ln(b_c)/n_m, x_c, l_c, n, L_a/l_k\}$

Choose 1 million random points in parameter space and find best fit to experimental data of crack speed vs. energy release rate.

Results:

{ $\tau \ln(b_c)/n_m$, x_c , I_c , n, L_a/l_k } = {-208.7 s, 8.9 μm , 4.155, 2.021, 1}

Crack speed from model and step loading experiments



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Crack propagation in short crack samples



- When the crack length is very short, pure shear calculation is not valid.
- Use FEM (Abaqus) to determine energy release rate, which for $\frac{a}{H} \ll 1$, is linear with crack length with a normalized slope of $\omega = 1.18 \pi$, thus for very short cracks, $G = \omega a W(I_1)$
- Well approximated by $G = G_{PS} \tanh(\frac{3.7a}{H})$ for longer cracks
- Relative to long starter cracks, need higher stretch to start short cracks, which then run into increasing G and accelerate until becoming unstable

Short crack experiments



Stretch and hold experiments performed for 4 initial
crack lengths and stretches
(a) 0.80 mm initial crack with stretch λ = 1.23,
(b) 0.75 mm crack with stretch 1.25,
(c) 0.49 mm crack with stretch 1.26,
(d) 0.34 mm crack with stretch 1.30.

Samples observed with camera and crack length measured

Sample images for 0.49 mm initial crack length with stretch 1.26

Testing model against short crack data

 Fit full FEM model results to exponential relationship between crack speed and energy release rate:

 $\frac{da}{dt} = v^* \exp\left(\frac{G}{G^*}\right)$ with $v^* = 2.144 \times 10^{-8} mm/s$, and $G^* = 12.896 \frac{J}{m^2}$

• Use $G = \omega a W(I_1)$, $\omega = 1.18 \, \pi$

 $\frac{da}{dt} = v^* \exp\left(\frac{\omega a W(I_1)}{G^*}\right),$

• Integrate with $a(0) = a_0$ to calculate crack length vs. time, modeling each experiment



Further experimental – model comparison

• From experimental data of crack speed

vs. G we find that for

$$v=0.25\,\mu m/s$$
 , $G=114.7\,\frac{J}{m^2}$

- Experiments with different initial crack lengths, stretching slowly until $v = 0.25 \ \mu m/s$.
- Record stretch ratio at which this occurs.
- For each crack length use FEM to compute stretch needed to reach

$$G = 114.7 \frac{J}{m^2}$$



Summary

- Delayed fracture in PDMS is studied experimentally for blunt notches and for sharp cracks under step stretch and constant stretch rate.
- In blunt notch sample crack grows after an extended hold
- In sharp, long crack samples loaded to G above threshold, crack grows at constant rate for step loading.
 - G vs crack speed is the same for step stretch and constant stretch rate
 - Crack speed, v, is observed to be an exponential function of energy release rate, G, in pure-shear (PS) sharp crack sample
- An analytic model based on chain Eyring type breaking kinetics is consistent with the measured relationship between G and V

