Equilibrium condition and physico-chemical effects of capillary condensation of water at crack tips in oxide glasses in moist air

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Slow Crack Propagation in Glasses

Stress-Corrosion
\( \sim \text{nm/s} \)

Critical Fracture
\( \sim \text{km/s} \)
Effect of temperature and humidity

\[ u = u_0 \exp(\beta K) = A \left( \frac{p}{p_0} \right)^m \exp \left( -\frac{Q_i - bK}{RT} \right) \]

\[ b = \frac{2V_a}{3\sqrt{\pi \rho}} \]

Wiederhorn, JACS (1967)
Wiederhorn and Bolz, JACS (1970)
Sample Geometry

DCDC
Double Cleavage Drilled Compression

\[ K_I = \frac{\sigma \sqrt{a}}{0.375 \frac{c}{a} + 2} \]

Dimensions:
4x4x40 mm³

Polishing:
Mechanical + CeO₂
RMS: 0.25 nm (10x10 µm² area)
Load Cell (Deben)

- Controlled atmosphere: $\text{N}_2 + \text{H}_2\text{O}$
- RH: $\sim 1\% \Rightarrow 80\% \pm 2\%$  $T: 22.0 \pm 0.5 \degree\text{C}$

Veeco
Dimension 3100
Nanoscope IIIa

‘Tapping’ mode
Slow Crack Propagation Observed by AFM
K-v curve

$v$ vs. $K_I$

$RH$ vs. $K$

$V$ vs. $K_I$

Time vs. $K_I$
Temperature anomaly on phosphate glass

Crichton et al, JACS (1999)
In-situ observation of a liquid condensate

Silica glass

\( v = 0.1 \text{ nm/s} \)

\( \text{RH} = 45\% \)

Wondraczek et al. JACS (2006)
Ciccotti et al. JNCS (2007, in press)
Energy dissipation due to the formation of a capillary neck between tip and sample

Nanometric water layer is both present on the glass surface and AFM tip

Hysteretic force-distance behaviour is responsible for a net energy loss at each contact

Zitzler et al., PRB (2002)
Capillary condensation

- Mechanical equilibrium: Laplace equation
- Thermodynamical equilibrium: Kelvin relation

\[ L_c \sim 1 \ \mu m \]

\[ L_c = f \left\{ \begin{array}{l}
\text{Critical distance: } H_c \\
\text{Crack profile: } 2u_y(X) \end{array} \right. \]

Irwin equation:

\[ u_y(X) = \frac{K_I}{E'} \sqrt{\frac{8X}{\pi}} \]
Calibration of the crack opening profile

1. FE Simulation

2. Reflection Interferometry

3. Irwin model up to 30 µm!

Two limit hypotheses

A) Slow evaporation: constant volume

B) Rapid evaporation: critical distance

Equilibrium
Experimental data support hypothesis B

The condensate length is determined by an equilibrium condition!

No apparent dependence on velocity!

Grimaldi et al. (2007, submitted)
Why is the critical distance so large?

VdW model for wetting surfaces

\[ H_c = 2 \, r_K + 3 \, e \]

\( r_K , e = f(RH) \sim nm \)

Effects to account for:

- Surface roughness
- Surface charge
- Hydration forces
- Ion exchange
- Impurities
Entailments

1. Kinetical

- Easier access of water molecules at the crack tip
- Reduction of region II plateau at high humidity

Wiederhorn, JACS (1967)
Entailments

2. Mechanical

\[ \Delta P \sim -100 \text{ atm inside the condensate (Laplace)} \]

- Closure effect
- Affect chemical reactions

3. Chemical

Corrosion + Ionic exchange

- Change in ionic concentration
  - pH
  - Wetting properties
Effect of pH on fracture in water

Silica glass

Sodalime

Wiederhorn et Johnson, JACS (1973)
Perspectives

A) New insights in the relation between the parameters of subcritical crack growth and

1) the environmental condition

2) the glass chemistry

B) Exploring the fracture surface chemistry through the effect on the wetting properties
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