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► **To cite this version:**

Priscille Fauvarque, Weiyang Feng, Laure Chomat, Daniel Bonamy, Fabrice Célarié, et al.. Subcritical crack growth of SiO₂-B₂O₃-Na₂O amorphous phase separated glasses. ICG Spring School 2024 - Glass for a sustainable future, Apr 2024, Lloret de Mar, Spain. . hal-04577425

HAL Id: hal-04577425

<https://hal.science/hal-04577425>

Submitted on 16 May 2024

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Subcritical crack growth of SiO₂-B₂O₃-Na₂O amorphous phase separated glasses

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Oxide glasses, commonly used in everyday life, have a major drawback: they have a brittle behavior. In a vacuum, abrupt failure occurs when the stress intensity factor (K) is greater than the fracture toughness (K_c). When exposed to the environment, small pre-existing flaws can grow even under relatively moderate stresses. This sub-critical crack growth is also commonly referred to as stress corrosion cracking (SCC).

Over the years, researchers have evidenced a clear dependence of crack velocity (v) as a function of K, with v depending on the temperature (T), relative humidity (RH) and chemical composition (CC) of the glass. Below the fracture toughness (K_c), three different regions have been identified, corresponding to different crack propagation mechanisms. Below a threshold limit, called the environmental limit, there is no crack propagation. In region I, the crack front velocity is controlled kinetically by the reaction between water and the stressed bonds at the crack tip [1]. Crack velocity (v) data follow Wiederhorn's exponential law with an apparent activation energy [2]. Data can also be fitted using Maugis power law. [3] Water diffuses towards the crack tip, and its time to reach the crack tip is the limiting factor in region II, leading to a plateau in the log^[7/6](v) vs. K curve. The crack velocity increases exponentially again with K in region III, which ends once K_c is reached [1].

Several studies have been carried out on subcritical crack growth in oxide glasses, but this phenomenon is less known for phase-separated glasses [4]. Recently W. Feng *et al.* studied amorphous phase separated (APS) SiO₂-B₂O₃-Na₂O glasses [4][5]. The objective of this work was to understand the influence of glass structure (S) on fracture properties. For this purpose, pristine glasses were compared to glasses of the same composition that went through different annealing protocols. These annealing protocols triggered secondary phase separation, greater than the rings. The size of demixed zones increases proportionally with the cubic root of the annealing time. How this secondary structure plays on the environmental limit and region I was investigated.

In our lab, we captured the v(T,H,CC,S) vs. K curves using double cleavage drilled compression (DCDC). Samples undergo SCC tests using a dual screw Deben machine in a well-controlled environment (T = 19 ± 1 °C ; RH = 40.0 ± 0.5 %). Crack growth is monitored by means of a tubular microscope and a LabVIEW program. Crack velocities (v) are obtained for region I and the environmental limit by post-processing images of the crack front position. Velocities correspond to a range between 10⁻¹¹ and 10⁻⁵ m/s.

To understand the link between the structure and fracture properties, additional tests were required. Atomic Force Microscopy (AFM) was used for post-mortem analysis of the fracture surface of the DCDC samples to capture the secondary phase size. Optical observations, X-ray diffraction (XRD) and Nuclear Magnetic Resonance (NMR) and Raman spectroscopies were also carried out to characterize the glass structure.

The poster will concern these previous works and will highlight future endeavors.

[1]: Wiederhorn, S.M. Influence of Water Vapor on Crack Propagation in Soda-Lime Glass. J. Am. Ceram. Soc. 50, 407-414 (1967). <https://doi.org/10.1111/j.1151-2916.1967.tb15145.x>

[2]: Wiederhorn, S.M. & Bolz, L.H. Stress Corrosion and Static Fatigue of Glass. J. Am. Ceram. Soc., 53, 543-548 (1970). <https://doi.org/10.1111/j.1151-2916.1970.tb15962.x>

[3]: Maugis, D. Subcritical crack growth, surface energy, fracture toughness, stick-slip and embrittlement. Journal of Materials Science, 20, 3041-3073 (1985). <https://doi.org/10.1007/BF00545170>

[4]: Feng, W. ; Bonamy, D. ; Célarié, F. *et al.* Stress Corrosion Cracking in Amorphous Phase Separated Oxide Glasses: A

Holistic Review of Their Structures, Physical, Mechanical and Fracture Properties. *Corrosion and Materials Degradation*, 2, 412–446 (2021). <https://doi.org/10.3390/cmd2030022>

[5]: Feng, W. Stress Corrosion Cracking of Sodium Borosilicate Amorphous Phase Separated Glasses. Ph.D.; Université Paris-Saclay (2022).