

Toughness and ultimate strength of amorphous silica identified through a combined microbeam and phase-field approach

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HIGHLIGHTS

- Microbeam experiments and phase-field modeling jointly give toughness and strength of amorphous silica.
- Notched and bone-shaped beams separate Griffith-driven from strength-driven fracture.
- The evaluated brittle properties of amorphous silica agree well with reported literature values.
- Notch geometry analysis provides practical guidelines for microbeam design.
- Small errors in geometry measurement cause large scatter in measured toughness.

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ABSTRACT

This work presents a new approach to evaluating the toughness, described by the critical energy release rate (G_c), and ultimate tensile strength (σ_c) of amorphous silica (SiO_2 glass), combining microbeam tests and phase-field calculations. The latter provides a numerical route to brittle fracture without prescribing explicit fracture surfaces *a priori*, enabling crack initiation and propagation to be tracked. Single-notched microbeams and newly designed bone-shaped microbeams with a notch-free gauge section were fabricated by Focused Ion Beam (FIB) milling and tested in bending in air, probing the brittle-fracture and strength-controlled regimes, respectively. Both geometries were modeled by Finite Element Analysis (FEA) coupled with a phase-field formulation. We found $G_c = 5.1 \text{ J/m}^2$ (critical stress intensity factor $K_{IC} = 0.61 \text{ MPa}\cdot\text{m}^{1/2}$), an intrinsic material length scale $\ell_c = 9.1 \text{ nm}$, and $\sigma_c = 6.8 \text{ GPa}$, consistent with previously reported brittle properties of silica glass. Through a parametric study, we quantified the impact of dimensional measurement error on the determined toughness. Unlike conventional micromechanical methods that yield only K_{IC} , our combined microbeam geometries and phase-field approach simultaneously deliver G_c and σ_c , bridging brittle-fracture characterization and the strength-controlled regime inaccessible to toughness-only techniques.

1. Introduction

The mechanical reliability of silicate glasses is undermined by their extreme surface sensitivity to flaws which act as stress concentrators triggering fracture before the structural element can macroscopically reach its intrinsic strength. As a result, the usual practical strength of glass falls several orders of magnitude below the theoretical strength predicted

from interatomic bonding considerations [1–3]. Therefore, predicting the fracture response of glass materials under real conditions is of utmost importance for the design of damage-tolerant glass products.

Silicate glasses are archetypal isotropic brittle solids. They behave in a clearly linear-elastic manner and fail catastrophically under tensile stress, although susceptible to environmentally assisted subcritical crack

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growth [4]. In the past, considerable effort has been devoted to measuring the brittle response of glass; however, very few works have directly quantified the fracture surface energy and intrinsic strength of glass. Wiederhorn's double-cantilever cleavage measurements [5] provided an estimate of fracture surface energy γ of a few J/m² for silicate glasses; Inagaki et al. [6] directly evaluated the work of fracture (*i.e.*, the fracture surface energy) from Chevron-Notched Short Rod (CNSR) experiments, later adapted by Lucas et al. [7] for direct evaluation of K_{IC} . Recently, Cui and Vinci [8] evaluated G_c from a combined FEA and bowtie-type Chevron notch microbeam approach. To et al. [9,10] evaluated G_c from the work of fracture (fracture surface energy) using CNB and K_{IC} using the Single-Edge Precracked Beam (SEPB), respectively. More recently, Rouxel et al. [11,12] reported the calculation of the fracture surface energy of glass from semi-ab initio considerations: average single bond energy and experimental density of glass. On the other hand, several works have evaluated G_c of glass indirectly from the measured K_{IC} values using Irwin's stress intensity factor relation [7,13–15]. It is noted that the value of K_{IC} remains valid as long as the experiments can ensure self-similar crack propagation in mode I loading at the critical state with a sufficiently large initial crack. In contrast, G_c is not constrained to the fracture mode because it arises from an energy balance between elastic strain and fracture energy. The intrinsic strength of glass has also been evaluated in silicate glasses, mainly using fibers; however, the literature remains limited to a few works, as in the case of G_c . Proctor et al. [16] reported the tensile strength of silica glass fibers tested by uniaxial tension in air. Weber et al. [17] evaluated the strength of mullite-composition glass fibers having a diameter of about 20 μm by applying pure uniaxial tension. Kurkjian and Tang [18,19] reported the evaluation of the ultimate strength of glass using different immersion liquids and temperatures through two-point bending tests of glass fibers. More recently, Brambilla et al. [20] and Luo et al. [21] evaluated the tensile strength of silica glass nanofibers by pure uniaxial tension in air and using in-situ TEM, respectively. Other evaluation methods include the ring-on-ring test, which usually yields much lower strength values than fiber-based evaluations [10,22] mainly due to the higher probability of finding critical surface defects in larger samples.

Over the last decades, the advent of FIB machining and micromechanical testing has moved these measurements to the micron scale, where flaw populations can be controlled and individual cracks sculpted. Recently, Li et al. [23] evaluated the fracture surface energy of single-crystal SiC using the double cantilever beam (DCB) approach, while Mueller and Žagar [24,25] evaluated K_{IC} of amorphous silica using chevron-notched microbeams with square and triangular cross-sections, respectively. Bruns et al. [26] estimated K_{IC} in amorphous silica using a micropillar splitting approach. Harding et al. [27] and Scholz et al. [28] evaluated K_{IC} from nanoindentation experiments in silica glass. Most of these works have focused on K_{IC} but not on G_c or the intrinsic strength of glass. This is mainly due to the lack of a consistent framework to numerically evaluate the experimental results, particularly for the evaluation of strength.

Due to a long-standing contradiction, neither fracture mechanics nor traditional strength-based verification can reliably predict failure. When a crack is present, the stress field becomes singular at the crack tip; thus, a strength-based criterion would predict a zero critical load. Conversely, if the sample is intact, fracture mechanics would predict an infinite critical load [29,30]. In practice, however, failure occurs regardless of the validity of either criterion. A solution to this problem was introduced by Leguillon [31] with the coupled criterion (CC). This criterion states that a crack may initiate and propagate over a finite distance only if two criteria are met simultaneously: 1) the stress exceeds a critical value (σ_c), and 2) the incremental energy release rate exceeds a critical value (G_c). This method has proven effective when the initial singularity and crack path are known *a priori*. A more contemporary approach is the phase-field method [32,33], which regularizes the stress field near the crack tip and enables the prediction of failure independently of the initial geometry.

The phase-field approach is rooted in Griffith's original concept [29], which states that a crack can propagate only if sufficient elastic energy is released from the body. Griffith postulated that creating a unit crack surface requires an energy expenditure of G_c , the critical energy release rate, an intrinsic material parameter. The phase-field formulation also introduces a second material constant (ℓ_c) that governs the extent of damage and, consequently, the degree of stress-field regularization. Although initially the length-scale served as a mathematical regularization construct for convergence of the energy functional [34], it was then reconsidered as an intrinsic property tightly connected to the material's strength in the phase-field formulation [35]. A material type vs. length-scale relation has been reported to be valid for a wide range of materials [36].

Several extensions of the classical variational phase-field approach have been proposed to improve the description of crack nucleation and material strength. In particular, cohesive phase-field formulations allow the tensile strength to be introduced and calibrated independently of the regularization length, notably through modified degradation and dissipation functions [37,38], while more recent variational formulations provide additional flexibility in prescribing material-specific strength surfaces [39,40]. Conversely, classical variational phase-field models intrinsically introduce a critical stress that depends on the fracture toughness and the regularization length ℓ_c , which can therefore be interpreted as a material characteristic length when fracture nucleation is strength-controlled [41–43]. In the present work, we adopt this latter interpretation and investigate whether, at the small scale considered here, a unique ℓ_c can be experimentally identified and directly related to the tensile strength. As shown below, the simplest phase-field formulation is sufficient to reproduce the experimental observations with a single set of G_c and ℓ_c values. Therefore, for the present application, introducing a more complex phenomenological formulation with an independently calibrated strength parameter is neither necessary nor desirable.

To achieve the evaluation of both G_c and ℓ_c , and ultimately σ_c , it is therefore necessary to design experimental setups that allow for independent calibrations and isolate the brittle-fracture from the strength-controlled regimes, combined with a numerical framework that does not presuppose an explicit crack path. In the present study, we design two complementary micron-scale geometries—a single-notched geometry and a newly designed bone-shaped geometry—obtained by FIB and tested under bending. These experiments allowed us to calibrate G_c and ℓ_c of amorphous silica, which gave access to the evaluation of σ_c . The geometries were modeled by finite element analysis coupled with a brittle-fracture phase-field formulation, which removes the need to prescribe the fracture surface *a priori*. In this work, the brittle-ductile phase-field formulation [44] was not used because we did not observe any plasticity in tension. The paper is structured as follows: Section 2 introduces the phase-field formulation and its two governing material parameters, describes the microbeam geometries, their FIB fabrication, and the bending test protocol. Section 3 presents the measured load–displacement responses together with the coupled FEA–phase-field analysis used to extract G_c , K_{IC} , ℓ_c , and σ_c . Section 4 discusses: a) the effect of crack geometry of notched microbeams on the fracture response, b) a parametric sensitivity study of the geometry on the evaluated G_c and stiffness, and lastly c) the connection between glass atomic structure and phase-field parameters.

2. Methods

In this section, we describe the experimental and numerical methods employed in this study. The experimental methods primarily focus on beam fabrication and testing, while the numerical methodology briefly outlines the well-known phase-field framework. Finally, we present the principal strategy for identifying phase-field fracture properties using the novel geometry proposed in this work.

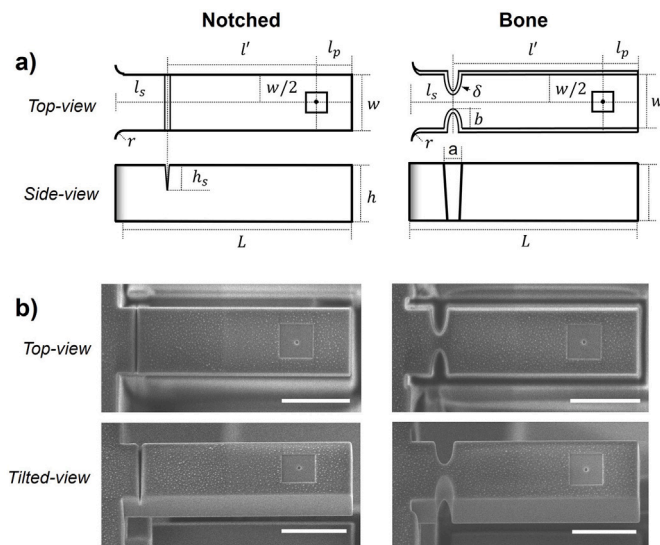


Fig. 1. a) Top- and side-view diagrams of the notched and bone microbeam geometries, and b) scanning electron microscope (SEM) images of the fabricated microbeams from both top and tilted observations. The white reference mark in the images corresponds to a length of 2 μm . (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

2.1. Microbeam fabrication

Silica glass (GE-124, <10 ppm OH) microbeams were fabricated through a FIB process (HITACHI FB2200, Japan) from Au-coated (10 nm) thin plates (5 mm $\times$ 5 mm $\times$ 0.5 mm) using an accelerating voltage of 30–40 kV and a beam aperture of 15–300 μm . The milling process started from one of the sample edges. Two types of beam geometries were fabricated: a single-notched geometry and a new bone-shaped geometry. Fig. 1 shows the schematics of the microbeams with associated geometric parameters. The summary of the geometrical parameters as measured from the SEM images is shown in Table 1. The geometry was measured from the SEM photos using image processing. The total length, width and height of the fabricated beams were close to those of the Chevron-notched microbeams reported by Mueller et al. [24] and provided a proper balance between FIB machinability and computational cost in terms of mesh density for the phase-field modeling.

The fabrication process was carried out in several steps, starting from rough milling (large aperture) and progressing to very fine milling (small aperture) for final tuning of the geometry. During the rough milling stage, significant re-deposition of material was observed on some of the microbeam faces, which required an intermediate milling step to remove the re-deposited zones.

For the notched microbeams, the notch had to be fabricated in multiple stages. First, the notch was milled from the top in a sufficiently thick cantilever, and then the outer faces were milled to eliminate the notch deflection caused by FIB milling near the microbeam's lateral surfaces [45]. The notch was fabricated at a voltage of 30 kV to create a wider top opening, allowing the milled material to leave the process zone more easily. The final tuning of the geometry for all microbeams was done using a small aperture size (e.g., 15 μm) and a high voltage (40 kV). This allowed us to obtain microbeams with almost straight faces and to reduce the amount of re-deposition.

For the bone-type beams, a single process (recipe) was used at a voltage of 40 kV and a beam aperture of 80 μm . Parameters a and b were chosen to be about 30–40% of the beam's width to ensure a clear differentiation from the fracture response of the notched beams and to ensure geometrical control. Parameter δ arose from the milling process itself due to the conical nature of the Ga^+ ion beam, thereby producing a taper angle. All the parameters were included in the FEA model to get a more precise approximation of the beam's fracture response.

2.2. Experimental loading

The load-displacement curves and fracture forces of the microbeams were measured in air (50% RH) using a nanoindenter fitted with a Berkovich indenter (Hysitron-Bruker Triboindenter TI950, USA). The indenter also serves as the tip for the Scanning Probe Microscope (SPM) integrated into the nanoindenter. The SPM allows detailed scanning of the sample surface at the nanoscale and precise positioning of the load. The TI950 indenter is an inherently force-controlled system operating through a capacitive transducer with a feedback loop (up to 78 kHz). However, all experiments were performed in displacement control mode. This particular mode is operated via a software-based feedback loop that uses real-time displacement data from the capacitive transducer to regulate the actuator's motion. Note that, upon sudden brittle failure, this experimental setup usually produces artificial displacement overshoots to compensate for the decrease in reaction forces. Glass plates were attached to thick steel disks with glue and left to cure overnight. A pre-load cycle to a maximum displacement of 50 nm was applied before the actual test to ensure appropriate contact. During the test, a maximum displacement of 500–1000 nm was applied at a loading

Table 1

Geometrical parameters for the notched (N) and bone (B) microbeams. All units are shown in micrometers. The parameter D corresponds to the notch tip diameter of the notched microbeams. All the parameters in the table correspond to those shown in Fig. 1a. The experimental uncertainty is $\pm 0.01 \mu\text{m}$ for all dimensional parameters except D , for which the uncertainty is $\pm 0.001 \mu\text{m}$.

	L	w	h	l'	l_s	h_s	l_p	D	δ	a	b	r
Sample	(μm)											
N-1	7.16	2.01	2.04	4.77	0.77	0.52	1.62	0.008	–	–	–	0.15
N-2	7.15	2.04	2.05	4.89	0.63	0.54	1.63	0.008	–	–	–	0.14
N-3	7.19	2.09	2.03	4.83	0.71	0.60	1.65	0.007	–	–	–	0.13
N-4	7.22	2.05	2.00	4.91	0.71	0.58	1.60	0.007	–	–	–	0.13
N-5	7.12	2.16	2.03	4.61	0.88	0.64	1.63	0.007	–	–	–	0.10
N-6	7.09	2.02	2.00	4.60	0.89	0.70	1.60	0.007	–	–	–	0.15
N-7	7.13	2.02	2.04	4.81	0.74	0.73	1.58	0.007	–	–	–	0.18
N-8	7.21	2.11	2.05	4.86	0.74	1.02	1.61	0.007	–	–	–	0.15
N-9	7.20	2.10	2.03	4.86	0.72	1.00	1.62	0.007	–	–	–	0.14
B-1	7.21	1.92	2.11	4.40	1.15	–	1.66	–	0.10	0.74	0.75	0.18
B-2	7.17	1.93	2.13	4.42	1.00	–	1.65	–	0.10	0.74	0.74	0.17
B-3	7.12	1.94	2.03	4.42	1.06	–	1.64	–	0.09	0.65	0.78	0.09
B-4	7.14	1.96	2.10	4.47	1.05	–	1.62	–	0.09	0.67	0.77	0.08
B-5	7.14	1.94	2.01	4.44	1.05	–	1.65	–	0.07	0.68	0.74	0.11

rate of 5 nm/s and an acquisition rate of 600 points/s. The resulting load-displacement curves were compliance and thermal-drift corrected via software.

2.3. Phase-field fracture method

The phase-field technique [32] is based on the reformulation of the original Griffith [29,30] theory on a variational basis [46]. The phase-field fracture model is based on a diffuse representation of localized discontinuities, where the crack surface is approximated by a continuous damage variable d ranging from 0 to 1. When $d = 0$, the material is fully intact, whereas $d = 1$ corresponds to complete failure, with total loss of stiffness and load-bearing capacity. This formulation enables a smooth transition from the undamaged to the fully fractured state, allowing the simulation of crack initiation and propagation without the need for explicit crack tracking. To calculate the new auxiliary variable, the total internal energy of the solid is minimized:

$$\Pi(\mathbf{u}, d) = E + W = \int_{\Omega} [g(d)\psi_0^+(\mathbf{u}) + \psi_0^-(\mathbf{u})] d\Omega + \int_{\Omega} \frac{3G_{c,pf}}{8\ell_c} [d + \ell_c^2 |\nabla d|^2] d\Omega \quad (1)$$

Here, E and W represent the strain and fracture energy components, respectively, and $\mathbf{u}$ denotes the displacement field defined over the domain Ω . The fracture energy is characterized by two key parameters: 1) the phase-field critical energy release rate $G_{c,pf}$ and 2) the crack surface density function. The damage diffusion is governed by ℓ_c and the spatial gradient of the damage ∇d in order to treat singularities in a thermodynamically consistent manner [47]. The undamaged strain energy density ψ_0 is decomposed into tensile (ψ_0^+) and compressive (ψ_0^-) parts:

$$\psi_0^{+,-} = \sum_{i=1}^3 \mu \langle \epsilon_i \rangle_{+,-}^2 + \frac{\lambda}{2} \langle \text{tr}(\epsilon) \rangle_{+,-}^2 \quad (2)$$

The eigenvalues, denoted as ϵ_i , are computed using the infinitesimal strain tensor, defined as $\epsilon = \nabla^S \mathbf{u}$. The material parameters λ and μ are the Lamé parameters. Functions $\langle \cdot \rangle_{\pm}$ stand for the positive $\langle x \rangle_+ = (x + |x|)/2$ and negative $\langle x \rangle_- = (x - |x|)/2$ parts.

In our formulation only the tensile component is subject to degradation through a quadratic function $g(d) = (1 - d)^2$. Consequently, only tensile energies contribute to crack evolution. To distinguish between tensile and compressive strain energies, we adopt the spectral decomposition proposed by Bernard et al. [48]. Finally, the total fracture surface Γ in the phase-field formulation is obtained by integrating the crack surface density over the domain:

$$\Gamma = \int_{\Omega} \frac{3}{8\ell_c} [d + \ell_c^2 |\nabla d|^2] d\Omega. \quad (3)$$

The mechanical and phase-field problems were solved using a staggered time-integration. To ensure irreversibility and to enforce positive damage evolution, Lagrange multipliers were implemented following the approach of Lu et al. [49]. All calculations used adaptive time stepping. Since our experiments revealed strains below 5%, a small strain formulation (small displacements and rotations) was adopted. For comprehensive implementation details, readers are referred to our recent work [47].

2.4. Correction of G_c based on the FEA mesh

It has been shown that brittle fracture phase-field calculations are subject to numerical errors. These numerical errors originate from: 1) time discretization, 2) spatial discretization. The first error has been minimized through the staggered phase-field algorithm as proposed in the literature [42,44]. The spatial discretization can be divided into two types: i) approximation error and ii) localization error. The first arises from the difference between the theoretical crack surface analytical solution (AT1 vs. AT2) and the FEA numerical approximation. The

second arises from the crack opening in the damaged state and is significantly larger than the approximation error. The spatial discretization error needs to be accounted for in the evaluation of the real material critical energy release rate G_c as shown by Molnár et al. [47], through the following relation,

$$G_c = G_{c,pf} \left(1 + \alpha \frac{m_s}{c_{\omega} \ell_c} \right), \quad (4)$$

where m_s is the size of the FEA mesh at the crack surface, and c_{ω} is a constant that depends on the damage model type (AT1 or AT2). In this work, we used the AT1 approximation with an elastic threshold. This model is consistent with a linear-elastic threshold before failure as observed in linear elastic brittle solids [35]. The parameter α describes the boundary conditions at the crack surface. In this work we used $\alpha = 1$ and $c_{\omega} = 8/3$, which correspond to the AT1 model without any boundary conditions. The FEA solid model was designed so that the ratio $\ell_c/m_s = 2$ (mesh size of one half the length-scale parameter), which ensures an approximation error of about 1% and a localization error of 18.1% [47]. For completeness, we performed a fracture-force mesh sensitivity study for both microbeam geometries. The results are shown in Fig. 8 in Appendix A. For a ratio $\ell_c/m_s = 2$, the calculations provide a suitable balance between accuracy of the predicted fracture forces, and computational costs; using finer meshes can lead to an increase in fracture force of about 5% compared to the selected mesh size ratio. In this manuscript we will refer to the corrected critical energy release rate simply as G_c .

2.5. Model and material parameter identification

The parameters G_c and ℓ_c were evaluated through a combination of two experiments: notched tests and bone microbeam tests, allowing both the brittle-fracture-dominated (notched) and strength-controlled (bone) fracture regimes to be probed.

The phase-field model requires the fracture energy G_c , the material's intrinsic length ℓ_c , and the elastic constants (E , ν) as input for each microbeam simulation. We determine G_c and ℓ_c using a non-iterative forward approach that returns, for every beam, the pair of parameters consistent with its measured fracture force. The inversion is exact, so the scatter in the resulting properties reflects only the differences in geometry and measured fracture force between individual experiments.

For a fixed geometry, elastic constants and mesh size, the critical (fracture) force P_c scales following Griffith's law as:

$$P_c = A(\ell_c) \sqrt{G_{c,pf}}, \quad (5)$$

where the pre-factor A depends only on the beam geometry, the elastic constants, and ℓ_c . A single FEA simulation with an arbitrary trial value of $G_{c,pf}$ therefore fixes A for a given ℓ_c and geometry. Equation (5) can then be inverted to give the fracture energy that reproduces the measured fracture force P_c^{exp} exactly:

$$G_{c,pf} = \left(\frac{P_c^{\text{exp}}}{A(\ell_c)} \right)^2 \quad (6)$$

Repeating this for a variety of ℓ_c values yields a continuous curve of $(G_{c,pf}, \ell_c)$ pairs, every point of which reproduces the fracture force of that particular beam. Finally, the parameter $G_{c,pf}$ needs to be corrected through Eq. 4.

The two geometries constrain this curve differently. In the notched beams the stress concentration is sharp (notch radius below 10 nm), and the fracture-force-consistent G_c is essentially independent of ℓ_c . In the bone-shaped beams the stress concentration is blunt and larger than the material length scale, so the fracture-force-consistent G_c depends strongly on ℓ_c . The intersection of the two curves in the G_c - ℓ_c plane therefore fixes both phase-field parameters for the material.

The stress intensity factor could alternatively be obtained from purely elastic FEA of the notched beams, by extracting nodal stresses or

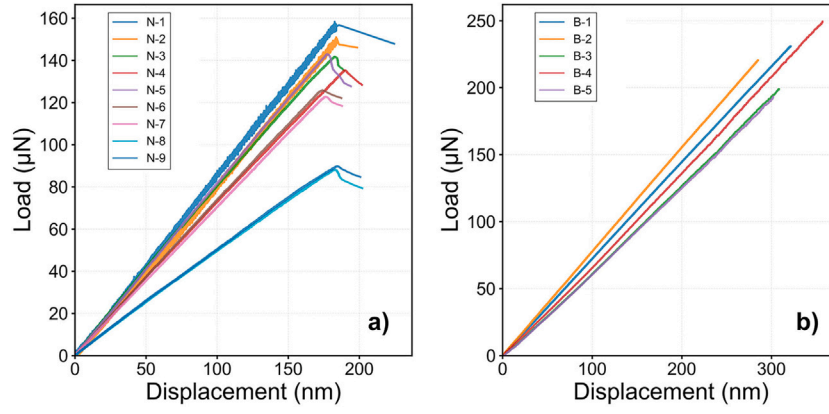


Fig. 2. Experimental load-displacement curves for a) notched and b) bone microbeams under a bending test. The maximum load for each curve corresponds to P_c .

displacements as a function of distance from the notch tip as described by Williams [50] and applied by Fujita et al. [51] in ion-exchanged glass microbeams. That route, however, gives no access to the ultimate tensile strength, which the present phase-field implementation does provide. Other methods, such as the PF-CZM, can introduce strength as a material property, but its value must be known *a priori* or determined through an inverse approach.

2.6. Evaluation of mode I stress intensity factor K_{IC}

From G_c it was possible to directly quantify K_{IC} for the case of single-notched microbeams using the plane strain assumption $K_{IC} = \sqrt{G_c E'}$, where $E' = E/(1 - \nu^2)$ is the effective modulus. The value of K_{IC} was also obtained for the notched beams using the relation proposed by Matoy et al. [15] for single-notched microbeams,

$$K_{IC} = \frac{P_c l'}{w h^{3/2}} f\left(\frac{h_s}{h}\right) \quad (7)$$

$$f\left(\frac{h_s}{h}\right) = 1.46 + 24.36 \left(\frac{h_s}{h}\right) - 47.21 \left(\frac{h_s}{h}\right)^2 + 75.18 \left(\frac{h_s}{h}\right)^3 \quad (8)$$

where P_c is the experimental fracture force of the notched microbeams. Eq. (7) is valid for $l_s:l':h:w = 2:5:1.1:1.7$ and for h_s/h ratios between 0.05 and 0.45.

2.7. Ultimate tensile strength evaluation

The ultimate tensile strength σ_c is defined as the maximum pure tensile stress that the material can withstand. In our numerical framework, σ_c is computed directly from the length-scale parameter ℓ_c . The homogeneous phase-field solution [47,52] provides the tensile strength σ_c of the material directly from the material parameters: Young's modulus (E), Poisson's ratio (ν), critical energy release rate (G_c), and the length-scale parameter ℓ_c ,

$$\sigma_c = \eta \sqrt{\frac{E G_c}{\ell_c}} \quad (9)$$

with the function η depending on the Poisson's ratio and the stress state (principal stress ratios). For the particular case of pure uniaxial tension, the principal stress ratios σ_2/σ_1 and σ_3/σ_1 are zero. Using the determined G_c and ℓ_c we computed the ultimate tensile strength of silica glass. Details of the numerical implementation can be found in previous work [42].

Table 2

Fracture forces of the investigated microbeam samples: Notched (N) and bone (B).

Sample	Fracture force P_c (μ N)
N-1	158.30
N-2	151.00
N-3	143.05
N-4	141.73
N-5	135.27
N-6	125.84
N-7	122.74
N-8	88.14
N-9	89.82
B-1	231.00
B-2	220.00
B-3	200.70
B-4	251.22
B-5	195.65

3. Results

3.1. Experimental results

In this section we present the experimental fracture responses of the silica microbeams including load-displacement curves, fracture forces, and fractography.

3.1.1. Load-displacement curves

The experimental load-displacement curves for all samples are shown in Fig. 2. For the notched microbeams, the fracture forces and elastic stiffness decrease with increasing notch depth h_s . For the bone-type microbeams, the fracture forces vary slightly among the samples (see details in Table 2). Overall, the fracture forces of the bone microbeams are about twice those of the notched microbeams.

All of the beams failed catastrophically after reaching a fracture load (P_c) and no subcritical crack growth was observed. The transducer of the indenter used in this work is inherently force-controlled; displacement is regulated via a software feedback loop and, therefore, it cannot follow the displacement when the crack proceeds.

3.1.2. Fractography

Fig. 3 shows an example of the scanning electron microscope observation of the microbeams after fracture. The notched microbeams show clear contrast between the fractured part and the initial notch, which allows the notch depth h_s to be determined more precisely. In the case of the bone beams, the cracks initiated at the narrow section of the beam. From the side-tilted observation, the cracks seem to have propagated at a slight inward angle from the initiation point.

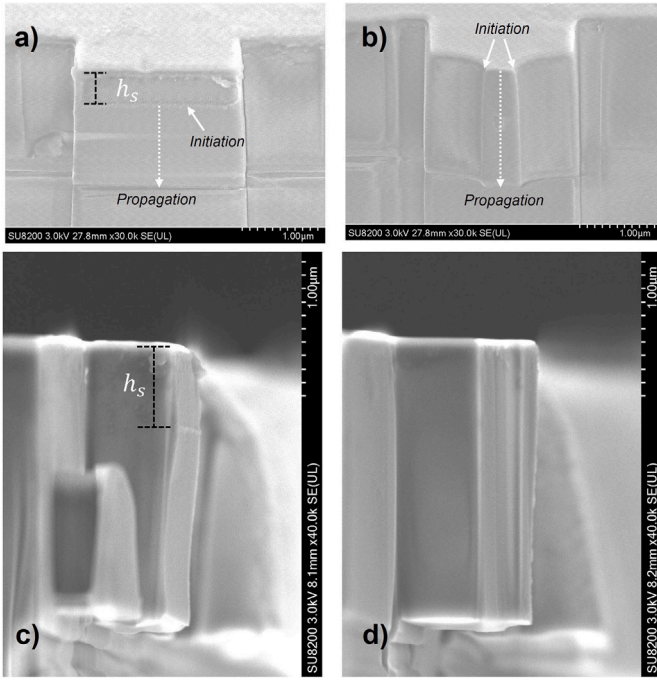


Fig. 3. Examples of scanning electron microscope (SEM) images of the broken notched (a and c) and bone (b and d) microbeams showing the fracture surfaces (black dashed lines). Also indicated are the notch depth h_s (frontal and side-tilted views), crack initiation zones (solid arrows) and crack propagation directions (dotted arrows). Notched and bone samples are N-6 and B-1, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3.2. Numerical results

In this section we show the numerical results of the investigation: FEA model, damage evolution, and material parameter identification.

3.2.1. FEA model

FEA simulations were performed in the ABAQUS 2022 FEA software. FEA solid models were built for both notched and bone geometries. For the case of notched microbeams (Fig. 4(a)), we used plane strain 2D models (quad CPE4 elements). For bone microbeams (Fig. 4(b)), we developed half-symmetry 3D models (4-node C3D4 tetra-elements). The core mesh region surrounding the beam's singularity was about 10 times larger than ℓ_c to ensure spatial accuracy of the damage variable d . ABAQUS Python scripts that adapt the core mesh size to the value

of ℓ_c were written for each microbeam geometry. This minimizes the numerical error of the damage variable d , as explained in Section 2.4. Bending simulations were performed using displacement control at the loading point of the microbeams with a maximum displacement of 1 μm . The damage field (d) was computed through a combined user-defined element (UEL) and user-defined material subroutine (UMAT). The numerical implementation of the subroutines is shown in detail in a previous work [47]. For the simulations, the elastic properties of silica ($E = 72$ GPa and $\nu = 0.17$) and diamond ($E = 1140$ GPa and $\nu = 0.07$) reported in our previous work [53] were used. The diamond indenter and the end part of the bone-microbeam apart from the neck region (Fig. 4(b)) were modeled as fully elastic to avoid phase-field convergence issues due to sharp contact nonlinearities.

Contact between the diamond indenter and the notched microbeam was modeled with the linear penalty method using a friction coefficient of 0.2. The mesh was locally refined to an element size of 10 nm over a 0.2 μm region around the loading point l_p (see Table 1). For the bone-shaped beams, contact was not modeled explicitly; instead, a displacement was enforced on a node set within a circular region of radius 0.2 μm centered on l_p , discretized with elements with an average size of 20 nm. In both geometries, a total vertical displacement of 1 μm was applied.

3.2.2. Damage evolution

One of the main advantages of the phase-field approach [47] is its ability to model crack initiation and propagation in a thermodynamically consistent manner, based solely on the competition between the elastic strain energy and fracture energy dissipation, without requiring explicit crack tracking or a predefined crack path. To illustrate this point, Fig. 5 shows calculation examples for the cases of crack initiation and propagation for both beam geometries along with their load-displacement curves. The results show the damage field at initiation and after propagation. For the case of the notched beam, the crack initiates and propagates off-axis at a slight tilt towards the base of the cantilever. This was indeed observed in the experiments (see Fig. 3). The slight tilt is due to the fact that the stress field has a slight shear component as shown previously by Molnár et al. [33] when testing fracture under pure uniaxial tension and pure shear. For the case of the bone beam, the fracture initiates at the edge near the singularity and propagates downwards and inwards simultaneously. In both cases the force decreases sharply upon fracture.

It should be noted that the post-initiation response differs between the experiments and the phase-field simulations due to the different loading conditions. Under the displacement-controlled conditions imposed in the simulations, the initial unstable crack jump is followed by a decrease in the energy release rate, allowing subsequent stable crack propagation. In the experiments, however, displacement

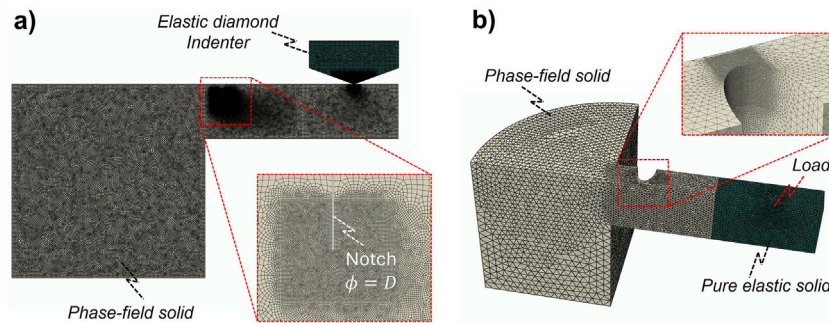


Fig. 4. Examples of FEA models used for the phase-field calculations using $\ell_c = 20$ nm: a) notched microbeams (2D - plane strain) showing the full model and a zoom of the notched area, and b) bone microbeams (3D - 1/2 symmetry) with a zoom of the singularity. The green (dark) areas are modeled as fully elastic while the gray areas are elastic-brittle phase-field elements. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

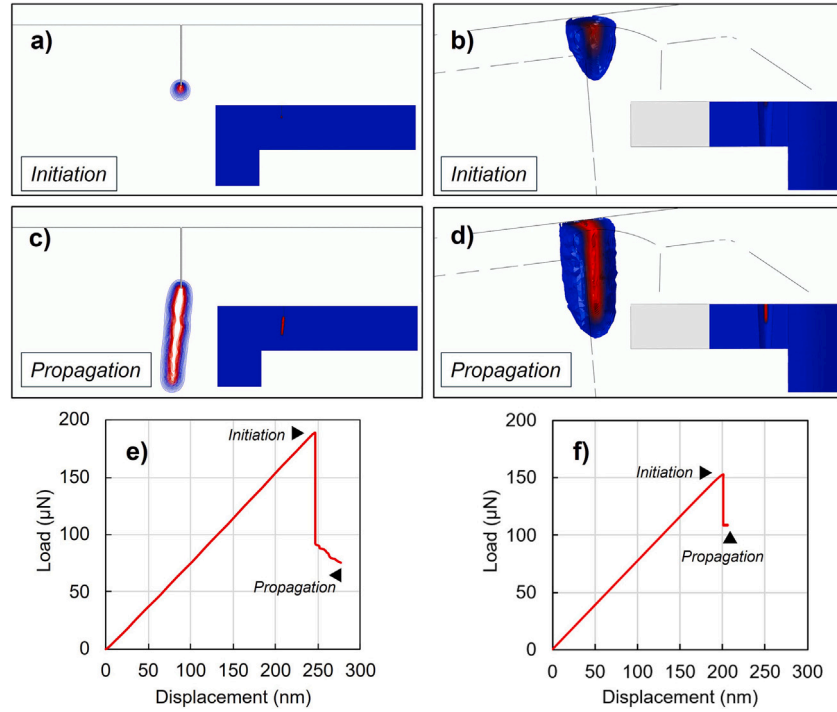


Fig. 5. Phase-field damage maps at the point of fracture initiation and after propagation along with their respective calculated load-displacement diagrams (bottom): (a,c,e) notched microbeams, and (b,d,f) bone microbeams. The damage field d varies between blue (no fracture, $d = 0$) and red (fully fractured, $d = 1$). Insets show the damage field in the whole model. For this example in both cases $G_c = 6 \text{ J/m}^2$ with length-scale parameters of $\ell_c = 50 \text{ nm}$ and $\ell_c = 100 \text{ nm}$ for the notched and bone geometries, respectively. A maximum vertical displacement of 1000 nm was applied in both beam geometries. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

control was imposed through a feedback loop on a force-controlled setup, whose response time was insufficient to capture this rapid transition. Consequently, the stable post-initiation propagation observed numerically (Fig. 5(e) and (f)) is not resolved in the experimental load-displacement curves.

3.2.3. Material parameter identification

The simulated fracture-force-consistent combinations of G_c and ℓ_c values for both notched and bone microbeam experiments are shown in Fig. 6. The figure shows the experimental variation bands and the median curve in each case. For the notched beam, the G_c values look constant even for different ℓ_c , while for the bone beam the value of G_c increases with the ℓ_c value. The intersection of the curves provides the optimal material parameters G_c and ℓ_c , with bounds defined by the black bars accounting for the experimental variation of both parameters.

From G_c we computed K_{IC} using the plane strain approximation. Lastly, from ℓ_c , we computed the ultimate tensile strength following Eq. (9). Tables 3 and 4 show the currently obtained results along with other reported data in the literature for silica glass. The estimated values of G_c , ℓ_c , K_{IC} , and σ_c for silica were 5.1 J/m^2 , 9.1 nm , $0.61 \text{ MPa}\cdot\text{m}^{1/2}$ and 6.8 GPa , respectively. In the case of G_c , Wiederhorn [5] reported a value of 8.7 J/m^2 for silica glass when tested in liquid N_2 and Cui and Vinci [8] suggested a G_c value of 5.2 J/m^2 for silica glass when tested in vacuum. The theoretical model by Rouxel [11,12] predicts a G_c of 7.3 J/m^2 for silica glass when using the single bond strength approximation. Overall, our results are within the literature range.

Regarding the length-scale parameter ℓ_c , previous literature shows smaller values compared to our results; however, those were estimated from theoretical considerations [54] or molecular dynamics simulations [55]; to our knowledge, the evaluated ℓ_c values are the first reported from experiments for silica glass.

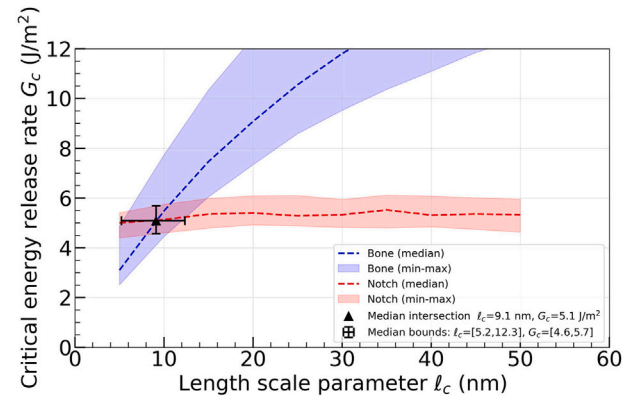


Fig. 6. Simulated fracture force-consistent G_c and ℓ_c for the notched (light red) and bone (light blue) microbeams. The optimal values correspond to the intersection of the median curves (dashed lines) and the variation is given by the bounds (black lines). The values at the intersection and their respective bounds for all the samples are shown in the legend. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The obtained values of K_{IC} for silica glass are within the range of literature values. We note that macroscopic experiments such as the SEPB method give consistently larger values, by about 10–20%, than those obtained from micromechanical methods such as the chevron-notched microbeam test [24,25], the micropillar splitting method [26], or the single-notched beam (current study). The K_{IC} values found through the analytical solution (Eq. 7) are larger than those obtained by our method, closer to those reported by macroscopic tests [7,13] and similar to those reported by Liu et al. [56] for silica microbeams using the

Table 3

Brittle fracture parameters for amorphous silica evaluated in air from notched and bone microbeam tests (this work), with the overall range of literature values (see Table 4). Individual literature sources are listed in Table 4. Values of K_{IC} in parentheses were obtained from the analytical solution (Eq. 7); all others are from phase-field calculations. The literature range includes results for tests in air and in vacuum.

Parameter	Unit	Median	Min.	Max.	Literature range
G_c	J/m ²	5.1	4.6	5.7	5.2–8.7
ℓ_c	nm	9.1	5.2	12.3	2.0–3.0
K_{IC}	MPa·m ^{1/2}	0.61 (0.75)	0.58 (0.72)	0.64 (0.78)	0.58–0.78
σ_c	GPa	6.8	5.3	9.1	5.1–13.0

same equation. It is noted that classical evaluations of K_{IC} and G_c using Linear Elastic Fracture Mechanics (LEFM) idealize the crack as infinitely sharp; however, in reality cracks may have a finite radius which might lead to an overestimation of the fracture surface energy inflating both K_{IC} and back-calculated G_c values. As the phase-field method regularizes the crack tip over the intrinsic length scale, it is expected to provide a closer representation of the fracture phenomena.

The evaluated strength values closely match the reported data for silica glass in air. The strength values reported by Tang et al. [19] in liquid nitrogen and by Luo et al. [21] in vacuum are about two times larger than those in air. This suggests that humidity may have a significant impact on the intrinsic strength of silica glass either through a reduction of G_c or an increase in ℓ_c at the surface. These strength values are expected to be larger when tested in inert environments possibly due to a “less water-modified” atomic structure at the surfaces. According to Eq. 9, water is expected to increase ℓ_c and/or decrease G_c of amorphous silica at the surface. It remains for future studies to evaluate the strength of glass in a vacuum.

Notably, FIB source ions can be implanted into the material’s surface over a distance of a few nanometers. For the particular case of notch fabrication (known as “overFIBing”), ion implantation has been reported [45]. To quantify the degree of ion implantation, in Appendix B we computed the Ga⁺ ion penetration depth in amorphous silica SiO₂ ($\rho = 2.21$ g/cm³) using the SRIM software [57]. The results are

summarized in Fig. 9. Two particular cases were computed: a) penetration of Ga⁺ ions when milling is performed at 40 kV almost parallel to the surface as in the cases of side trimming or bone-type beams, and b) penetration of Ga⁺ ions when milling is performed at 30 kV perpendicular to the surface as in the case of notched beams (overFIBing). The estimated penetration depths were ~ 10 nm and ~ 30 nm for the first and second case, respectively. Similar Ga⁺ penetration depths have been reported in other amorphous systems such as amorphous carbon [58], epoxy [59], and polydimethylsiloxane (PDMS) [60] although they differ in their chemistry. These values are comparable to the evaluated ℓ_c of amorphous silica. Yet our obtained strength/toughness values are consistent with those reported in the literature using Ga-free samples. The effect of Ga⁺ ions on the mechanical properties of glass remains unclear and the literature on the topic barely exists. A systematic assessment of Ga⁺ ion-damage effects on the glass structure and properties for example through classical molecular dynamics (MD) simulations remains a worthwhile direction for future investigations.

Finally, it is relevant to point out that the results from our material parameter identification may change depending on the selected phase-field functionals (e.g., crack density function, degradation function, and split). The appropriate selection of phase-field functionals depends strongly on the material type and stress state. For a perfectly linear-elastic brittle material such as amorphous silica, we found that an AT1 model with an elastic threshold was able to capture the elastic-brittle nature of silica glass. In contrast, models such as AT2 start damaging (losing stiffness) as soon as a load is applied, also resulting in a softening tail after the critical failure. Such behavior is more appropriate for ductile materials for which cubic type degradation functions have also been proposed. The split describes how the elastic strain energy is degraded with loading. In this work we considered that glass only fractures/damages under tension. Under compression, oxide glasses densify and undergo plastic flow (homogeneous and/or localized). Only localized plastic flow is known to induce damage in silicate glasses [61]. In such cases other theories such as gradient plasticity may account for plastic damage [62].

In the next section we will discuss the role of the beam geometry in the fracture response and the effect of measurement error on the

Table 4

Reference data for amorphous silica brittle fracture parameters, with testing conditions and methods. DCC – double-cantilever cleavage; CNMB – chevron-notched microbeam; PF – phase-field; MD – molecular dynamics; NB – notched-beam; CNSR – chevron-notched short rod; NI – nanoindentation; PS – pillar splitting; NMB – notched microbeam; 2PB – two-point bending (fibers); ROR – ring-on-ring. † Back-calculated from the reported $K_{IC} = 0.62$ MPa·m^{1/2} via $G_c = K_{IC}^2 (1 - \nu^2)/E$ ($E = 72$ GPa, $\nu = 0.17$); the original paper reports K_{IC} , not G_c . ‡ Values measured on nanofibers with diameters below 5 nm.

Parameter	Value	Condition	Method	Author	Ref.
G_c (J/m ²)	8.7	liquid N ₂	DCC	Wiederhorn	[5]
	7.3	–	theoretical	Rouxel & Yoshida	[11]
	5.2 [†]	vacuum	CNMB (bowtie)	Cui & Vinci	[8]
ℓ_c (nm)	3.0	–	PF (theoretical)	Tomić et al.	[54]
	2.0	–	MD	Molnár & Barthel	[55]
K_{IC} (MPa·m ^{1/2})	0.73	dry air	NB	Smith & Chowdary	[13]
	0.78	air	CNSR	Lucas et al.	[7]
	0.65	vacuum	CNMB	Mueller et al.	[24]
	0.67	vacuum	CNMB	Žagar et al.	[25]
	0.58	air	NI	Harding et al.	[27]
	0.68	vacuum	PS	Bruns et al.	[26]
	0.60	air	NI	Scholz et al.	[28]
	0.71	vacuum	NMB	Liu & Shinozaki	[56]
σ_c (GPa)	5.9	air	μ -fiber tension	Proctor et al.	[16]
	6.0	air	2PB	Baikova et al.	[63]
	5.1	air	2PB	Tang et al.	[19]
	0.14	air	ROR	To et al.	[10]
	11.5	liquid N ₂	2PB	Tang et al.	[19]
	~ 10	air	nano-fiber tension	Brambilla & Payne	[20]
	10–13 [‡]	vacuum (TEM)	nano-fiber tension	Luo et al.	[21]

estimated G_c values. Finally, the connection between brittle properties and the atomic structure of silica will be discussed.

4. Discussion

In the previous section, the brittle properties of silica glass were successfully evaluated through the combined microbeam test and phase-field simulations. The results were consistent with previous literature and provide guidelines for the evaluation of the brittle behavior of other amorphous materials. In this section we discuss the following topics: a) the role of microbeam geometry (notch depth h_s and crack tip diameter D) in the fracture response (fracture force), b) the effect of errors in geometry measurements on the estimated G_c values, and finally c) the connection between the brittle properties and structure of silica glass and possible implications for silicate glasses.

4.1. Role of microbeam's geometry in fracture response

We performed a parametric study on notched beams by changing the notch geometry (depth and tip diameter) and material toughness (G_c). This analysis provides information on how the geometry affects the fracture responses (brittle vs. strength regimes).

Fig. 7(a) shows the fracture force as a function of the notch depth normalized by the total height of the beam. For very small notch depths ($h_s/\ell_c < 1$) with a crack tip radius smaller than ℓ_c , the fracture force is independent of the notch depth; this corresponds to the strength regime, in which the material fails upon reaching its tensile strength. When the depth of the notch exceeds $h_s/\ell_c = 1$, the fracture force decreases with notch size at a rate of -0.5 on a logarithmic scale, corresponding to the Griffith brittle fracture regime. This regime persists up to an h_s/h ratio of about 0.4. At larger depths, the relationship between the fracture force and depth becomes highly non-linear due to boundary effects (bottom side of the beam); in this region the stiffness of the beam decreases rapidly with notch depth. In our current investigation, the values of h_s/h were between 0.25 and 0.5, and were therefore near the boundary between the Griffith and non-linear brittle regimes. The equation proposed by Matoy et al. for the evaluation of K_{IC} (Eq. (7)) using geometrical considerations of the microbeam is valid for h_s/h values between 0.05 and 0.45. The values of K_{IC} obtained from G_c through phase-field calculations (plane strain approximation) are valid only as long as we are in the brittle Griffith regime.

In multiple materials, size-dependent transitions can be explained using higher-order theories such as non-local, strain gradient or couple stress models. The proposed phase-field method (although it is not a higher-order model) demonstrated that the transition between the Griffith and strength regimes could be effectively represented through the introduction of a material's length-scale parameter. Maranganti et al. [64] evaluated strain-gradient elasticity constants in silica glass and other materials using atomic-scale simulations. In their work it was found that the relevant length scales for strain-gradient elasticity were of the order of 5 Å, much smaller than continuum scale lengths and not relevant for our case.

The relationship between the fracture force and notch diameter (normalized by ℓ_c) at constant notch depth h_s is shown in Fig. 7(b). The fracture force P_c is constant as long as the diameter D is smaller than ℓ_c . It is noted that the fracture force slightly increases when the tip radius approaches zero as is assumed in Linear Elastic Fracture Mechanics (LEFM). When the notch diameter goes beyond ℓ_c , the fracture force increases and material failure cannot be described by Griffith fracture. Finally, the fracture forces for a constant, sufficiently large notch depth (h_s) follow a linear relationship with G_c in a log scale with a slope of 0.5, in accordance with Griffith's law (Fig. 7(c)).

These results provide guidelines for the geometrical design of the microbeams. In this sense, by controlling the geometry we can predict whether the microbeam will behave as a Griffith brittle solid or will fracture through strength-controlled failure.

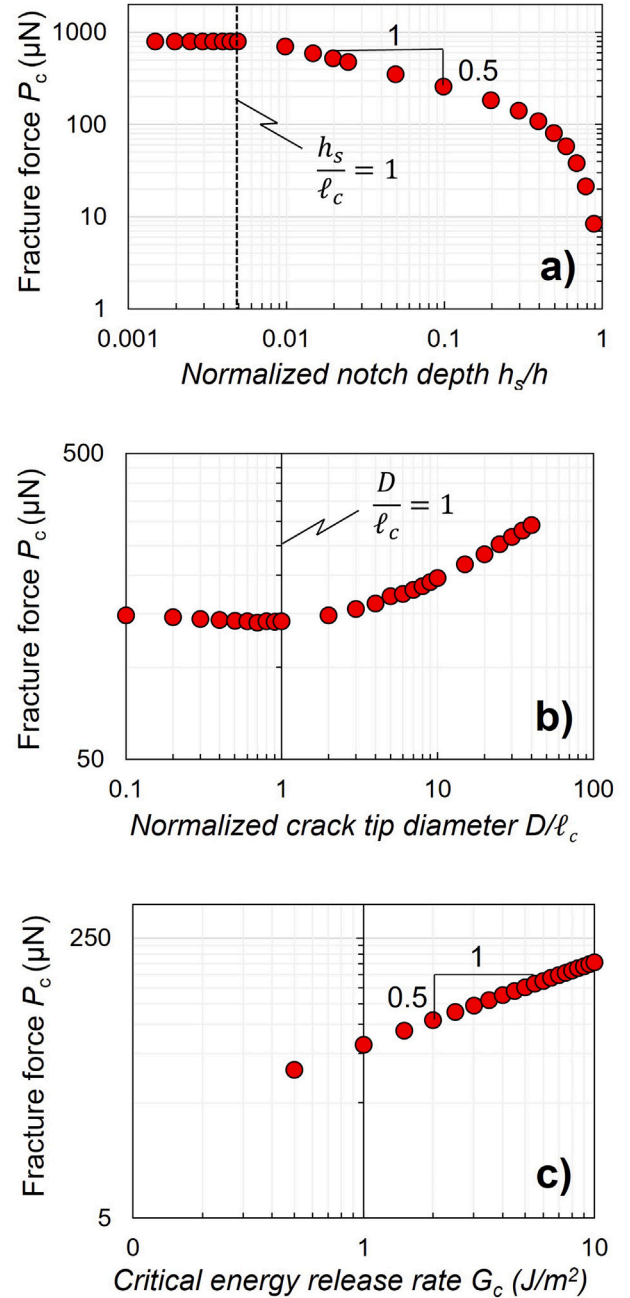


Fig. 7. FEA phase-field-computed fracture forces P_c of a notched microbeam: (a) effect of the notch depth normalized by the total beam height h , (b) effect of the notch diameter normalized by the length-scale parameter ℓ_c , and (c) effect of the critical energy release rate. The curves were built using a beam with initial dimensions the same as those of sample N-6. For cases (a) and (b), $G_{c,pf} = 6$ J/m² and $\ell_c = 10$ nm.

4.2. Effect of geometry measurement error on the evaluation of G_c

Errors in the geometrical measurements of the cantilevers can result in significant errors in the evaluation of their brittle properties (G_c and K_{IC}) and stiffness. Mueller et al. evaluated the effect of errors in geometry measurements on the resulting errors in stiffness and fracture toughness of Chevron notched microbeams [24]. Their analysis suggests that errors in the measurements of the height (h) as well as the width w of the notches have the most significant effect on the evaluated stiffness and fracture toughness. In their work, an error of 5% in those geometrical parameters can result in a 10–30% error in estimated

Table 5

Effect of 5% incremental change in the geometrical parameters (L , w , h , l_s , h_s , l_p , δ , a , b) and Young's modulus E on the percentage (%) variation in estimated critical energy release rate G_c and elastic stiffness (values in parentheses). Reference geometrical parameters and fracture forces have been used for each microbeam type.

Type	L	w	h	l_s	h_s	l_p	δ	a	b	E
Notched	19 (–15)	–9 (4)	–45 (50)	–2 (1)	6 (–2)	–4 (3)	–	–	–	–4 (5)
Bone	17 (–14)	–15 (6)	–15 (12)	–2 (1)	–	–4 (4)	–1 (1)	–5 (–1)	19 (–2)	–5 (5)

stiffness and fracture toughness. This behavior is expected since the stiffness of microbeams with fixed cross sections is proportional to the cube of their height ($\propto h^3$) and to their width ($\propto w$). We conducted a similar analysis on the investigated microbeam geometries. The results are summarized in Table 5. For the notched beams, the most significant effect on the evaluation of G_c and stiffness originates from errors in the measurement of the microbeam height h , followed by its length L and width w . For the bone beam, similar trends to the notched case are observed; however, the strongest influences on both G_c and stiffness are given by parameters L and h followed by w . The height has less influence in the case of the bone geometry compared to the notched microbeams. We define the stiffness here as the elastic slope of the load-displacement curve during the bending of the beams as evaluated by the FEA calculations. It is noted that for the bone beams, errors in the geometry measurements of the narrow section can also have a strong impact on the calculated G_c , particularly the parameter b . The effect of errors in the elastic modulus remains proportional in the evaluation of both G_c and stiffness for both geometries.

4.3. Brittle properties of amorphous silica glass and connection with its atomic structure

Our evaluations of the brittle properties of silica glass reveal that it is a material with multi-GPa strength and a damage regularization size (ℓ_c) of a few nanometers. This length-scale goes beyond the local structure (coordination polyhedra) and medium range order (polyhedra connectivity) of silica glass. Recent work by Molnár and Barthel [55] showed through atomic-scale simulations of silica glass that the fracture surface energy γ comprises a contribution from the free surface energy and a major contribution (four times larger) from damage, defined as the local reduction in the material's stiffness without plasticity. The diffuse damage could extend over 2 nm from the crack surface inside the material, correlating spatially with the number of broken rings in the structure. In addition, they confirmed that the ℓ_c obtained from the homogeneous phase-field solution was close to that obtained through MD calculations. Our results show that the diffuse damage region is within a few nanometers, similar to that found in MD simulations. Although MD simulation results are strongly dependent on the choice of semi-empirical potential, our results support the view that ℓ_c is not just a numerical parameter but connected to the material's atomic structure.

The effect of tensile stress on the fracture response and atomic structure of silicate glasses has been evaluated in the past. Taniguchi and Ito [65] showed by molecular dynamics simulations of a soda-lime-silicate glass under uniaxial tension that the structure exhibits three main changes: expansion of T-O-T angles, changes in ring statistics (small rings form at the expense of larger rings), and growth of cavities. Tamura et al. [66] evaluated the uniaxial tension behavior of silica glass from first-principles calculations where they found that rotations of SiO_4 tetrahedra serve as the main stress release mechanism during elastic tension. Pedone et al. [67] investigated the structural changes of silica under tension using different boundary conditions. During the straining process, both the Si-O distances and Si-O-Si angles increase linearly until the failure point. In parallel, the ring distribution becomes broader, enhancing the formation of both small and large rings at the expense of medium-sized rings, and is followed finally by void coalescence and fracture. It was noted that crystalline SiO_2 (cristobalite) has an intrinsic strength of about three times that of its glass counterpart, attributed to its single-sized ring distribution compared to silica glass, suggesting a smaller length-scale. As shown by Molnár and Barthel [55],

the ring changes seem to be closely related to the damage of the material. Du and Cormack [68] showed in sodium silicate glasses that increasing the amount of sodium at the expense of silica promotes the formation of large-membered rings at the expense of the main 6-membered rings observed in silica glass. Therefore, in glasses with a large amount of non-bridging oxygens or network modifiers and broad ring distributions, damage is expected to be more diffuse than in regular-sized ring structures such as those of crystalline SiO_2 or silica glass.

Since ℓ_c governs the spatial extent over which the elastic energy is dissipated through bond rupture resulting in stiffness reduction, materials whose networks accommodate stress through the rearrangement of a broader distribution of ring sizes are expected to exhibit a more delocalized (diffuse) damage zone, and therefore a larger ℓ_c . Following Eq. (9), where the strength is inversely proportional to the square root of ℓ_c and proportional to the square root of G_c , this larger length-scale translates directly into a reduced intrinsic strength σ_c . Amorphous silica, with its comparatively narrow ring-size distribution and highly connected tetrahedral network, thus represents one limiting case in oxide glasses of a well-regularized structure with a small ℓ_c and consequently a high intrinsic strength. It is important to note that the fracture surface energy γ contributes to the ultimate strength of the material through G_c . For the particular case of silica, both the Si-O bond density and the high bond energy contribute to the large G_c of amorphous silica [11,12].

This structural interpretation, although consistent with our observations, remains qualitative at present. It therefore remains for future work to evaluate, for oxide glasses, the effects of different network formers such as boron oxide (B_2O_3), phosphorus oxide (P_2O_5), or aluminum oxide (Al_2O_3), as well as the role of network modifier type and non-bridging oxygens, on the length-scale ℓ_c of the glass, helping to establish a more accurate description of brittle fracture and strength across glass families. Such a framework would ultimately allow the brittle response of a glass to be anticipated from its composition and short- to medium-range order, providing a rational basis for the design of stronger and more damage-tolerant glass compositions. Finally, although amorphous silica was completely brittle in tension, the partial presence of plasticity under tension in other silicate glasses or brittle materials remains possible.

5. Summary and conclusions

In this work we presented a new approach for the evaluation of the brittle properties of amorphous silica: the critical energy release rate G_c , the intrinsic length-scale ℓ_c , the critical stress intensity factor K_{IC} and the ultimate tensile strength σ_c . The approach combines microbeam bending experiments with phase-field fracture calculations. The phase-field method uses a thermodynamically consistent formulation based on the direct transformation of elastic energy to fracture surfaces enabling tracking of crack nucleation and propagation from singularities without requiring an explicit fracture surface to be prescribed *a priori*. The main conclusions are summarized as follows:

- Two complementary FIB-fabricated geometries were tested on amorphous silica, each probing a different fracture regime. Single-notched microbeams, whose sharp singularities emulate the Griffith brittle regime, allowed the determination of G_c , which describes the material's toughness, independently of ℓ_c . The newly designed bone-shaped microbeams with blunt singularities provided access to the strength-controlled regime, enabling the evaluation of the material's intrinsic length ℓ_c and, therefore, of the

ultimate tensile strength σ_c . This approach extends brittle-fracture characterization beyond toughness-only approaches commonly used in micromechanical studies.

- The intersection of the fracture-force-consistent G_c - ℓ_c curves from both geometries yielded, for amorphous silica, $G_c = 5.1 \text{ J/m}^2$, $K_{IC} = 0.61 \text{ MPa}\cdot\text{m}^{1/2}$, $\ell_c = 9.1 \text{ nm}$, and $\sigma_c = 6.8 \text{ GPa}$. These values agree closely with previously reported data. To our knowledge, this is the first experimentally derived regularization length-scale for amorphous silica.
- A parametric study clarified how the notch depth and tip radius govern the transition between the strength-controlled and Griffith brittle regimes, providing practical guidelines for the geometrical design of microbeam experiments.
- In agreement with previous works by Mueller et al. [24], errors in dimensional measurements (particularly of the beam height h , width w and length L) translate into substantial uncertainty in the determined G_c and stiffness, highlighting the importance of accurate geometric characterization.
- Beyond amorphous silica, the proposed methodology offers a general framework for simultaneous evaluation of toughness and strength in brittle solids, including metallic glasses, polymers, ceramics, or other amorphous systems.

CRediT authorship contribution statement

Gustavo Alberto Rosales-Sosa: Writing – original draft, Visualization, Validation, Software, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Gergely Molnár:** Writing – review & editing, Supervision, Software, Methodology, Conceptualization. **Yoshinari Kato:** Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Conceptualization. **Taichi Nakashima:** Writing – review & editing, Methodology, Investigation. **Takahito Ohmura:** Writing – review & editing, Supervision, Resources, Methodology. **Etienne Barthel:** Writing – review & editing, Supervision, Methodology. **Guillaume Kermouche:** Writing – review & editing, Supervision, Methodology. **Sergio Sao Joao:** Writing – review & editing, Methodology. **Shingo Nakane:** Writing – review & editing, Supervision, Project administration, Funding acquisition. **Hiroki Yamazaki:** Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Fracture-force mesh sensitivity study of notched and bone microbeams

We performed a fracture-force mesh sensitivity study for the notched and bone microbeams. For this study we used the geometries of beams N-6 (notched) and B-4 (bone); the reader is referred to Table 1 of the manuscript for the geometrical parameters of each beam. In both cases, the values of G_c and ℓ_c were set to 5.1 J/m^2 and 10 nm , respectively. Several ratios of length-scale to mesh-size (ℓ_c/m_s) were tested, ranging from 0.33 to 10.0. For the bone microbeams, the maximum ratio was set to 3.33 due to computational limitations. The $G_{c,pf}$ values used in the FEA simulations were obtained from the mesh-size corrected G_c using Eq. 4 as described in Section 2.4. Fig. 8a and b show the results for the notched and bone beams, respectively. For ratios smaller than 2, the fracture force diverges quickly from the mesh-independent fracture force. A ratio $\ell_c/m_s = 2$ ensures a proper balance between fracture force accuracy and computational cost. By using a finer mesh, the predicted fracture force increases by about 5% compared to that with the selected mesh size ratio.

Appendix B. Prediction of Ga⁺ ion beam penetration in amorphous silica using the SRIM software

This section shows simulations of the penetration of Gallium ions during the FIB process using the SRIM software [57]. For the simulations we selected Ga⁺ ions accelerated at 40 kV (89.9° incidence angle) and 30 kV (0° incidence angle) following the trimming/bone and notch making processes, respectively. The simulations were performed using a total number of 10,000 Ga⁺ ions. The target was modeled as SiO₂ with a density of 2.21 g/cm^3 . The material width and height were set to 2000 nm. The simulations predicted collision trajectories of Ga⁺ ions with the target at a single surface point. The results are shown in Fig. 9a and b. The predicted weighted average depths $\langle y_{ion} \rangle$ were 8.2 nm and 27.5 nm for the $\theta = 89.9^\circ$ and $\theta = 0^\circ$ cases, respectively. When the beam hits perpendicularly to the surface, the ions reach deeper but a surface ion depletion region is observed. On the contrary, when the beam is almost parallel to the target, the penetration is shallower but ions concentrate more near the surface.

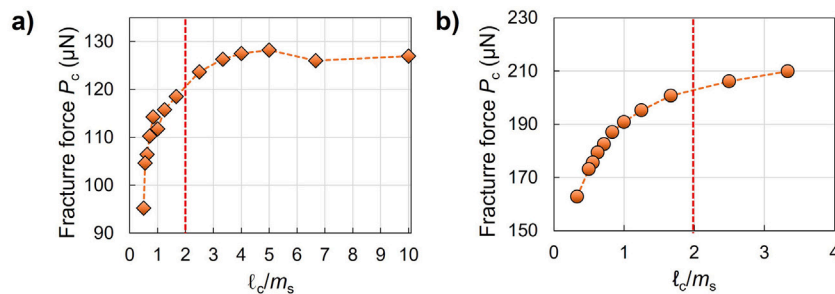


Fig. 8. Fracture force mesh sensitivity study of the notched (a) and bone (b) microbeams. The fracture force is plotted against the ℓ_c/m_s ratio. The red-dashed lines show the ratio used in this work for the evaluation of the G_c and ℓ_c material parameters ($\ell_c/m_s = 2$). For each plot, we assumed a constant $G_c = 5.1 \text{ J/m}^2$ and $\ell_c = 10 \text{ nm}$. The value of $G_{c,pf}$ in each case was computed using Eq. 4. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

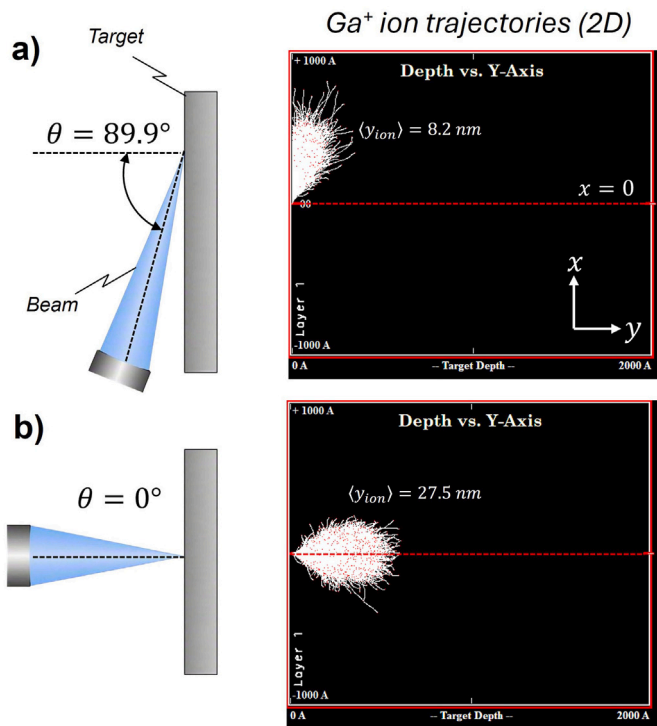


Fig. 9. Ga^+ ion trajectory simulations with an SiO_2 target: a) 40 kV beam with an incidence angle $\theta = 89.9^\circ$ (almost parallel to the target surface), and b) 30 kV beam with an incidence angle $\theta = 0^\circ$ (perpendicular to the target surface). The images show the ion trajectories in the x–y space. The field of view is 200 nm $\times$ 200 nm. The weighted average depth $\langle y_{ion} \rangle$ is also shown.

Data availability

Data will be made available on request.

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