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Short communication

# A direct correlation between damage parameters and effective permeation coefficients in composite laminates

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ABSTRACT

We introduce an innovative approach for determining the gas permeability of composite laminates, explicitly accounting for inter-fiber fracture. Our method forges a direct correlation between the Continuum Damage Mechanics (CDM) damage parameter for transverse inter-fiber fracture and the effective permeation coefficients, which are crucial in assessing leak tightness. This correlation stems from a geometric similarity between the ratio of the damaged material's load-carrying capacity to that of its pristine state, and the relative projected crack length as crucial parameter for the effective permeability assessment. This CDM-based approach represents a significant advancement in directly deriving a laminate's permeability from mechanical failure analysis results. This is essential for the design process of Type V hydrogen storage tanks. Literature-based experimental results validate the plausibility of our method, proving its effectiveness across various laminate orientations and damage scenarios. Nonetheless, the observed deviations highlight the need for detailed damage information, elaborate material characterization.

## 1. Introduction

In Type V hydrogen tanks, the load-sustaining composite laminate also serves as the critical permeation barrier, ensuring leak tightness, which is significantly influenced by micro-damage within the laminate [1]. Notably, inter-fiber cracking markedly increases the laminate permeability [2] even though it is hardly relevant to the mechanical performance. This type of damage is inevitable in lightweight hydrogen tanks, which are typically subjected to both cyclic thermal and cyclic mechanical loading [3–5]. Thus, a comprehensive permeation assessment, focusing on damage effects, is essential in the design and operation of these tanks.

One approach to describe the effective permeability/diffusivity $l_{eff}$ of a composite ply is the homogenization of the fiber–matrix composite [6] including damage in form of inter-fiber fracture. To evaluate the leakage $J$ for a given concentration gradient over thickness $\frac{\Delta c}{t}$, Fick's law (Eq. (1)) is employed, which suitably characterizes gas diffusion through a laminate in many cases [7]. The homogenization according to Ebermann et al. allows to express $D$ by the effective value $l_{eff}$.

$$J = -D\,\frac{\Delta c}{t} \tag{1}$$

Damage initiation in the individual plies of a composite laminate is predicted through the evaluation of failure criteria [8]. Cracking in form of a particular failure mode is expected to evolve in the composite as soon as the corresponding failure criterion is fulfilled. Progressive damage modeling through Continuum Damage Mechanics (CDM), as pioneered by Kachanov [9,10] and by Chaboche [11], is a widely used method for modeling stiffness degradation in finite element (FE) analyses. Matzenmiller's application of CDM to orthotropic materials [12] has made this approach available for analyzing damage in composite materials at both meso- and macro-scales. CDM is a widely used method for modeling stiffness degradation in FE analyses (e.g. [13–16]) also with regard to the analysis of hydrogen tanks [17,18].

In this study, we aim to establish a method to calculate a composite laminate's gas permeability on the basis of a mechanical progressive damage analysis employing Continuum Damage Mechanics (CDM). Following the methodology outlined in Ebermann's approach [6], we accomplish this by systematically analyzing and correlating the geometric variables from CDM-based damage assessments with the key parameters that influence permeation in damaged composites. This approach provides a novel framework for predicting and evaluating the permeability of composite laminates in practical engineering applications, especially in the context of hydrogen storage and transport.

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## 2. Methodology

### 2.1. Continuum damage mechanics for inter-fiber fracture

The CDM relates the loss of load sustaining area with an integral stiffness degradation of the material. Voids evolving on the micro scale cause of this area loss. According to Eq. (2), the ratio of the remaining load sustaining cross section $A_e$ and the initial cross section $A_0$ is expressed as the difference of one and a damage variable $d \in [0,1]$, where $d = 0$ represents the pristine condition and $d = 1$ a fully developed crack.

$$(1-d) = \frac{A_e}{A_0} \tag{2}$$

The true stress $\sigma$ in the remaining load-sustaining cross section $A_e$ is expressed as an effective stress $\hat{\sigma}$ referring to the initial cross section $A_0$. To calculate the effective stress, we apply Hooke's law to the true stress and replace the ratio of cross section $A_e/A_0$ by $(1-d)$, as the Eqs. (3) and (4) show.

$$A_0\hat{\sigma} \quad = \quad A_e\sigma \tag{3}$$

$$\hat{\sigma} = \frac{A_e}{A_0}\sigma = (1-d)\,\sigma = (1-d)\,E\varepsilon \tag{4}$$

The Eq. (4) is the baseline for multiple state-of-the art damage models for composite materials [17,19,20]. Damage in a unidirectional ply requires several damage variables $d_i$ to be calculated for the degradation of different stiffness components. An inter-fiber crack through the thickness is commonly represented by $d_2$, directly affecting the transverse modulus $E_{22}$, and the shear moduli $G_{12}$, $G_{23}$. The constitutive law to calculate $d$ depends on the damage-propagating forces – cyclic, creep, or quasi-static loading – and requires a damage evolution law.

A common damage evolution for FE applications is the strain-driven bi-linear law according to Eq. (5) as proposed by Mi et al. [21]. $d(\varepsilon)$ is calculated from damage initiation strain $\varepsilon_0$ and the ultimate failure strain $\varepsilon_1 = \frac{2G_{Ic}}{X_{22t}b}$ defined by the critical energy release rate $G_{Ic}$, the transverse tensile strength $X_{22t}$, and the characteristic length $b$. Another typical variant is an exponential damage evolution described by Eq. (6) with Eq. (7) as an adaption of Maimi's proposal [14].

$$d_{bilin}(\varepsilon) = \frac{\varepsilon_1}{\varepsilon_1-\varepsilon_0}\left(1-\frac{\varepsilon_0}{\varepsilon}\right) \tag{5}$$

$$d_{exp}(\varepsilon) = 1-\frac{\varepsilon_0}{\varepsilon}\exp\left[\alpha\left(1-\frac{\varepsilon}{\varepsilon_0}\right)\right] \tag{6}$$

$$\alpha = \frac{2bX_{22t}^2}{2E_{22}G_{Ic}-bX_{22t}^2} \tag{7}$$

### 2.2. Effective permeation coefficients accounting for damage

Effective permeation coefficient boundaries for a pristine composite are derived through the homogenization of the fiber–matrix composite, as detailed by Ebermann et al. [6].

For a scalar concentration gradient, scalar analytical Hashin–Shtrikman (HS) bounds [22] of the effective/homogenized permeability are obtained through Eq. (8) which are applicable under the assumption of isotropically arranged isotropic phases, in our case the fiber reinforcement and the polymer matrix. In the equations, $l_j$ represent the permeability of the matrix and the fiber, and $v_j$ the respective volume fractions. The HS variational approach compares the composite's inhomogeneous permeability $l$ with the permeability $l_0$ of a hypothetical homogeneous body under identical boundary conditions. The effective permeability bounds $l_{HS}$ in (8) are derived by varying $l_0$ to the extreme values. Given that the phases are isotropic, the permeabilities can be expressed as scalars. The upper bound is achieved when $l_0$ is minimal compared to $l$ and the lower bound when $l_0$ is maximally large. These scalar bounds provide a quantifiable measure of the composite's effective permeability. $\delta_{dim}$ is the dimensionality and $l_0$ is either set to the matrix or the fiber permeability to obtain the upper or the lower bound, respectively. The leakage assessment based on this homogenized permeability can be conducted by evaluating Fick's law given in Eq. (1).

$$\begin{aligned} l_{\mathrm{HS}} &= l_0 - (1+w\underline{l})^2 a + \underline{l}^2 w + 2\underline{l} \\ \underline{l} &= (a^{-1}-w)^{-1} \\ a &= \sum v_j b_j \\ b_i &= (w + (l_j - l_0)^{-1})^{-1} \\ w &= (\delta_{dim}\, l_0)^{-1}. \end{aligned} \tag{8}$$

For permeability analysis of a cracked composite we consider an arbitrary inter-fibre crack as shown in Fig. 1. Here, $a_c$ is the crack length in the 23-plane. Gas flow/concentration gradient is also assumed in thickness direction (3-direction). The vital influence of such a crack to the effective permeability is accounted for by a two-staged stacking of material phases:

First, the homogenized composite ply and the leak path through a crack form a parallel stack characterized by the effective void volume fraction $v_{void}$ (Eq. (9)). In Eq. (9) $l_{HS}$ denotes the permeability boundaries of the pristine material and $l_{void}$ the hydrogen permeability within the void volume. As considering void volume fraction, the corresponding permeability $l_{\mathrm{HS,void}}$ has to be a function of the crack opening displacement (COD) and the crack density $\mu_{cr}$. Second, a series stack of a cracked material volume with $l_{\mathrm{HS,Cracked}}$ and a pristine material volume with $l_{\mathrm{HS}}$ represents the permeability of a (partially) cracked ply calculated by Eq. (10). The length the crack projected to the permeation direction (ref. Fig. 1) is obtained through the dot product of $\mathbf{a}_c$ and the normalized concentration gradient $\frac{\nabla \mathbf{c}}{|\nabla \mathbf{c}|}$. The normalized projected crack length $\tilde{a}_{proj}$ is obtained by dividing the projected crack length $a_{proj}$ through the ply thickness $t$ (ref. Eq. (11)).

$$l_{\mathrm{HS,void}} = \left(1-v_{void}\right) l_{\mathrm{HS}} + v_{void}\, l_{void} \tag{9}$$

$$l_{\mathrm{HS,CrackedPly}} = \frac{1}{\frac{\tilde{a}_{proj}}{l_{\mathrm{HS,void}}} + \frac{1-\tilde{a}_{proj}}{l_{\mathrm{HS}}}} \tag{10}$$

$$\tilde{a}_{proj} = a_{proj}\cdot\frac{1}{t} = \frac{\mathbf{a_c}\cdot\nabla\mathbf{c}}{|\nabla\mathbf{c}|\ t} \tag{11}$$

To calculate the effective permeability of a multidirectional laminate $l_{eff}$ containing n plies, the permeability of each individual ply $i$ is assessed for its respective crack length $\tilde{a}_{proj_i}$ and then combined in a series stack, as detailed in Eq. (12). This approach calculates the laminate's overall permeability by considering the contributions of each layer's permeation resistance and is applied by various researchers [23,24].

$$l_{\mathrm{eff}} = \frac{\sum_i^n t_i}{\sum_i^n \frac{t_i}{l_{\mathrm{HS,CrackedPly}}\left(\tilde{a}_{proj_i}\right)}} \tag{12}$$

### 2.3. Correlating CDM with effective permeation coefficients

In CDM, the damage variable $d$ is derived by a comparison of the pristine cross section $A_0$ and the effective load-carrying cross section $A_e$ (ref. Eq. (2) and Fig. 1). The difference between these areas (Eq. (13)) defines the projected crack surface $A_{c2}$, oriented normal to the 2-direction and the effective stress $\hat{\sigma}$. Consequently, the damage variable $d_2$ is determined by the ratio of this projected crack area to the original cross section $A_0$. By assuming a crack that extends through the whole width of the material, where $A_{c2} \propto w$, this ratio can be simplified to a ratio of lengths in the 3-direction, as detailed in Eq. (14). The resulting fraction is then equivalent to the projected crack length $\tilde{a}_{proj}$, which

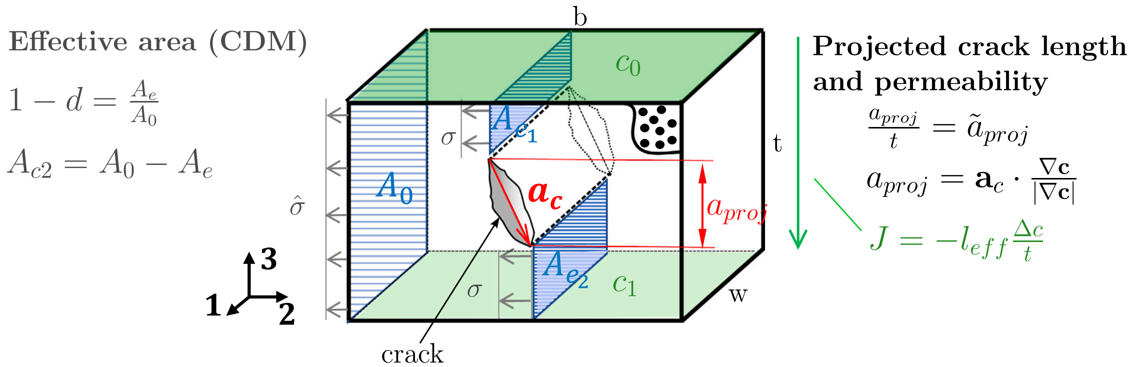



**Fig. 1.** Geometric derivation of the CDM damage variable $d_2$ (left) and the relative projected crack length $\tilde{a}_{proj}$ for the calculation of an effective permeability (right).

is a key parameter in calculating the material's effective permeability through Eq. (10).

$$A_0 - A_e = A_0 - \sum_i A_{e_i} = A_{c2} \tag{13}$$

$$d_2 = \frac{A_0 - A_e}{A_0} = \frac{A_{c2}\frac{1}{w}}{A_0\frac{1}{w}} = \frac{a_{proj}}{t} = \tilde{a}_{proj} \tag{14}$$

The validity of Eq. (14) vitally depends on the assumption of through-the-width cracking, which implies crack propagation in the ply's thickness direction. This phenomenon has been demonstrated through experimental investigations by Saito et al. [25] and numerical studies by Arteiro et al. [26]. Given this established understanding, the assumption of through-the-width cracking is plausible and justified.

## 3. Results and discussion

The correlation established in Eq. (14) enables the evaluation of a composite laminate's permeability by substituting the normalized projected crack length $\tilde{a}_{proj}$ by the CDM damage variable $d_2$ in Eqs. (10) and (12). In structural analyses, $d_2$ is derived from the progressive damage law, allowing for a direct correlation between external strain levels and permeability. This is demonstrated by applying the approach to different examples from the literature. Hamori et al. [27] provide pertinent experimental data on the permeability of cross-ply laminates of T700SC/2500 under increasing bi-axial loading. To evaluate the mechanical response and to determine the damage variable $d_2$ depending on the bi-axial load, the elasticity ($E_{22} = 9380\,\text{MPa}$ [28]), the strength ($X_{22t} = 63\,\text{MPa}$ [28]), and the fracture toughness ($G_{Ic} = 0.25\,\frac{\text{mJ}}{\text{mm}^2}$ [27]) of the material are used.

In cases where experimental data does not include strain-dependency, a meaningful comparison can still be made if information about laminate cracking is available. Effective laminate permeability, is obtainable by setting the CDM damage state for cracked plies $d_2 = 1$, while uncracked plies are considered with $d_2 = 0$. Saha et al. [29] report normalized permeability for stitched and unstitched specimens from two different materials after thermal cycling. The cracking state is reported for each tested configuration after certain cylce intervals.

Fig. 2 compares the normalized permeability with experimental results of the normalized leak rates the two aforementioned cases. The strain-dependent results in Fig. 2(b) depict the mechanical degradation for both bilinear and exponential constitutive laws and the respective influence to the dimensionless permeabilities $\bar{l}_{eff}^{bilin}(d_{bilin})$ and $\bar{l}_{eff}^{exp}(d_{exp})$ that are obtained through normalization of the calculated permeabilities with the value for the undamaged composite ($\bar{l}(d) = \frac{l_{eff}(d)}{l_{eff}(d=0)}$). The indices *exp* and *bilin* indicate the value obtained through the exponential and the bilinear CDM degradation laws. The results reveal a quantitatively plausible progression of the strain (and damage) dependent permeability. Additionally, the permeability curve's slope indicates that an exponential damage evolution model is capable to characterize the damage-dependent permeability more accurately than a bilinear model. The fact that Hamori et al. conducted their experiments under cryogenic conditions might also contribute to the good agreement of the results, as the current version of our model calculates the permeability for open cracks.

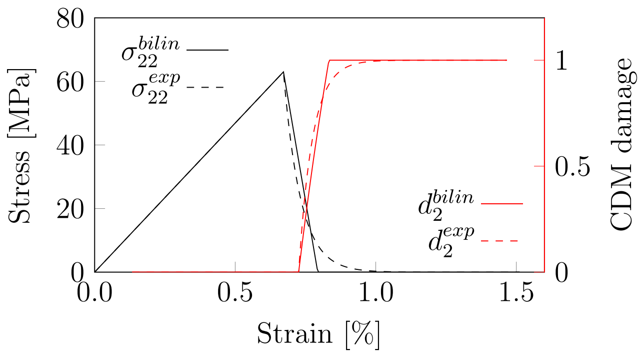
The results displayed in Fig. 2(c) only allow to assess the effect of entirely cracked plies to the permeability of a laminate, as the permeability is not recorded throughout the damage evolution. Saha et al. presented results across two material configurations (SE and IE) for stitched (S) and unstitched (U) laminates. To maintain consistency and clarity in our comparative analysis, we have retained the original nomenclature used by Saha et al. in presenting the results depicted in Fig. 2(c). As the crack density in the plies the midspan values reported by Saha et al. were used.

Relative to the findings of Hamori et al. the increase in permeability observed in the data from Saha et al. is significantly lower. This disparity arises because, in Hamori's experiments, all plies of the laminate are subjected to uniform damage. Conversely, in Saha's studies, only a few plies are affected by cracking, resulting in a less substantial permeability increase. While the overall damage-driven permeability increase is in a plausible range, also some deviations are found. Notably, the calculation underestimates the effect of the damage to the permeability of the $[45, -45]_{4S}$ laminate, in particular for the SE-S material. In Saha's study, damage detection relies on edge observations of the specimens, which may not have captured local damage occurring deeper within the laminate. The underestimated permeation is likely attributable to these undetected and hence unreported cracks in other plies of the laminate.

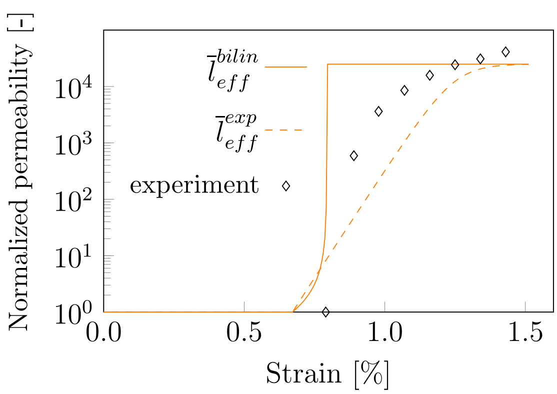
An underestimation of the permeability is also reported by Schultheiss in his doctoral thesis about the hydrogen diffusivity in an undamaged composites. He traced that effect back to the presence of "highly diffusive paths in carbon fiber reinforced plastics" [30]. This perspective is further supported by the recent work of Katsivalis et al. [31], whose experimental results provide evidence for significant effects of manufacturing defects and initial cracks in the specimens enhancing the permeability. As a further possible enhancement factor, nano-porosity was identified through electron microscopy. These observations collectively suggest that initial material defects could significantly impact the permeability of composite materials, independent of any mechanical damage.

### 3.1. Limitations and future challenges

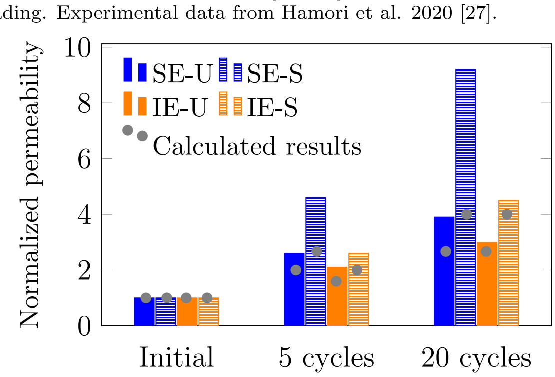
While the results affirm the suitability of the CDM-based permeability evaluation, they also highlight significant challenges in analyzing




(a) Damage evolution of a unidirectional ply in the $[0_4, 90_4]_{2s}$ stack employed in the experiments of Hamori et al. 2020 [27].




(b) Normalized permeability of a $[0_4, 90_4]_{2s}$ stack under biaxial loading. Experimental data from Hamori et al. 2020 [27].




(c) Normalized permeability of a $[45, -45]_{4S}$ stack from two different materials with and without stitching after thermal load cycles. Experimental data from Saha et al. 2021 [29].

**Fig. 2.** Diagrams displaying the calculated permeability alongside experimental results for three cases obtained from the literature.

composite laminates' leak tightness. The deviations observed underscore the difficulty of conducting accurate permeation assessments without comprehensive material characterization. Consideration of the initial void content is crucial to determine effective permeabilities for pristine materials. Moreover, understanding the impact of loading on both pristine and damaged states is essential.

Furthermore, a strong correlation between strain and leak rate in cracked composite laminates has been observed by Grenoble and Gates [32], Yokozeki et al. [33] or by Kumazawa et al. [23], which can be attributed to the COD – an effect that is not yet sufficiently addressed through Eqs. (9) and (10) and not directly obtainable from the CDM constitutive law. The literature [34,35] reports a near linear relationship between the COD and the strain applied perpendicular to the crack, expressed by COD $= a_v\varepsilon + b_v$. Taking into account the crack density $\mu_{cr}$ this allows for a correlation of the effective void volume content $v_{void}$ with the strain expressed through (15). Using this relation in Eq. (9) considers the COD in the permeability calculation of a cracked composite ply.

$$v_{void}\left(COD(\varepsilon), \mu_{cr}\right) \propto \mu_{cr} \cdot \left(\varepsilon a_v + b_v\right) \quad (15)$$

Furthermore, the presented method only accounts for damage induced by mechanical load. With regard to an actual tank structure, the experimental characterization of the initial void content would be a great enhancement to assess the permeability.

## 4. Conclusion

We presented a direct correlation between the continuum damage parameter and the effective permeability. This correlation enables the direct determination of hydrogen permeability from the results of a mechanical failure analysis, offering a streamlined and efficient process for evaluating the leak tightness of Type V hydrogen storage tanks. The comparison with experimental data confirmed the plausibility of the approach and also pointed out the issues to be address to achieve a comprehensive leakage assessment, accounting for all relevant effects. These are:

- The effective void content $v_{cr}$ as the key parameter of the permeability of a damaged material $l_{\text{HS,Crack}}$ has to be correlated with the COD and the crack density. Also the experimental characterization of the void content in a real structure is of importance to reliably determine the laminate permeability.
- The effective permeability of an undamaged material ($d = 0$) must to be reliably determined, considering the influence of temperature and the as-built condition.
- The role of the ply interfaces as a permeability barrier and the crack intersections as leak paths has to be addressed.

Beyond refining the model itself, a critical next step is to apply the derived equations to a real-world tank scenario. This can be achieved by integrating our model with a thermo-mechanical fatigue model that employs CDM for material degradation.

### CRediT authorship contribution statement

**Raffael Bogenfeld:** Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Caroline Lüders:** Writing – review & editing, Supervision, Investigation. **Michael Ebermann:** Writing – review & editing, Investigation, Conceptualization. **Vineeth Ravi:** Writing – review & editing, Resources, Investigation, Formal analysis.

### 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.

### Data availability

The data used in the article is taken from the literature.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