Fracture of corrosion protecting layers: investigation by indentation test

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Keywords: pipelines, corrosion, coatings, fracture.

Abstract. The mechanical properties of protective coatings produced by corrosion on the internal surfaces of pipelines transporting hydrocarbons have been investigated by indentation tests, which have revealed the spall or cracking attitude of the film. The role of material properties controlling the coating integrity and the main damaging mechanisms have been identified with the aid of the numerical simulation of the experiment.

Introduction

Pipelines transport worldwide large volumes of strategic resources. Consequently, procedures and practices to monitor their integrity represent challenging investigation areas for operators, especially in the hydrocarbon sector. The presence of water and of chemical species such as CO_2, H_2S, O_2 represents prominent factors causing internal corrosion, which is one of the most active and dangerous damage mechanisms, which can lead to reduction of pipe wall thickness and ultimately to leak or burst failure.

CO_2 corrosion is described by predictive models, mainly based on empirical correlations with laboratory and/or field data [1]. Other analyses have been based on a strict mechanistic analysis of the various processes involved in acid corrosion of carbon steel. However, in spite of the huge amount of available literature and collected failure data, it is reasonably easy to understand a corrosion event “backward” with failure analysis methods while a large degree of uncertainty is associated to the attempt of predicting future damage. This limitation is partly connected to the lack of reliable information about the material properties, which may degrade during the life-time of the pipeline, for instance under the action of hydrogen produced by corrosion reactions and absorbed by the metal [2].

Indentation test has been recently proposed as a suitable tool to determine the cross-thickness mechanical characteristics of pipelines steel [3]. The same testing procedures have been also exploited to investigate the properties of the thin layers produced by corrosion on the internal surfaces of the pipes. The results obtained during a preliminary explorative investigation campaign carried out on carbon dioxide (CO_2) or hydrogen sulphide (H_2S) coatings have been presented in [4].

The interpretation of the experimental output can benefit from the results of a simulation model of the indentation test, which is presented in this contribution.

Experiment

In this preliminary explorative campaign, samples have been produced in a reactor in controlled environment at different pressure, in the range 1÷170 bar, producing corrosion coatings of different thickness and morphology. All specimens have been subjected to indentation tests at different maximum load, up to 200 N, by rounded conical (Rockwell) and sharp pyramidal (Vickers) tips,

which may return complementary information. Representative indentation curves are for instance shown in Fig. 1 and compared with those relevant to the bulk material (non-treated, NT).
The jumps of the penetration depth observed in some situation reveal the spall or cracking attitude of double layer corrosion film and the possible penetration of fracture into the bulk material. These phenomena have been evidenced by SEM analysis of the indented surfaces and of the cross surfaces produced by careful cuts through the indentation marks. Meaningful micrographs are documented in [4].

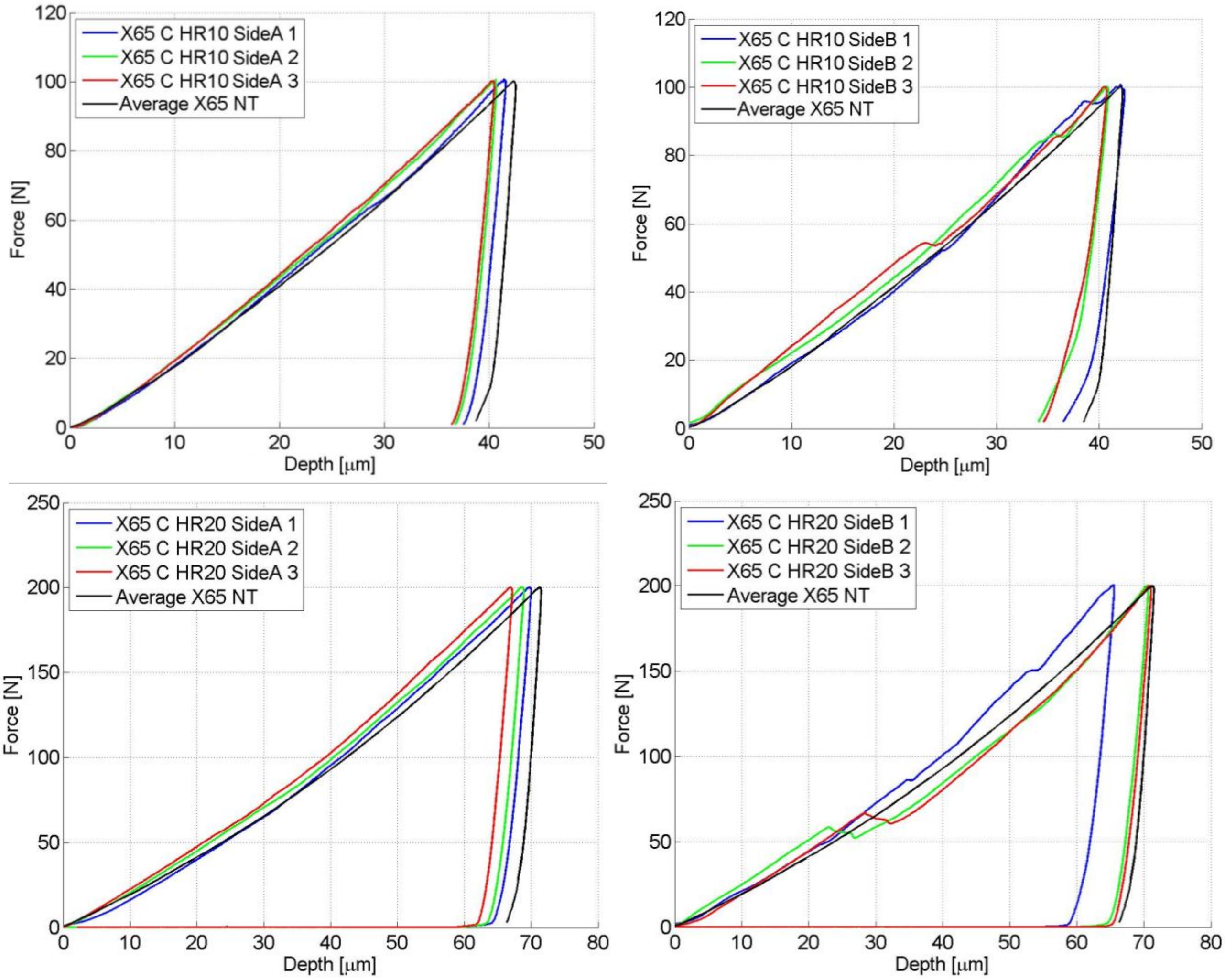

Fig. 1. Indentation curves relevant to carbonated steel specimens – Rockwell tip: single corrosion layer on the left; double on the right.

Model

The interpretation of the experimental results produced by the performed indentation tests can benefit from the simulation of the experiment, which can contribute to identify the role of the material properties controlling the coating integrity and the main damaging mechanisms.
Simulations were performed by a commercial finite element (FE) code [5], implementing models of a representative portion of the bulk material covered by a layer of corrosion products. The steel was described by the classical Hencky–Huber–Mises (HHM) model with exponential isotropic hardening and assumed mechanical properties listed in Table 1. These values were inferred from the reference indentation curves, relevant to the tests performed on uncoated metal samples, see Fig. 1.
The mechanical response of the coating was assumed to be governed by the associative Drucker–Prager (DP) criterion with linear isotropic hardening rule. The onset of irreversible plastic deformation is thus defined by the condition:

$$\sqrt{\frac{1}{2}\sigma'_{ij}\sigma'_{ij}}+\alpha p-k-h\lambda=0 \tag{1}$$

where: σ'_{ij} denote the components of the deviatoric stress tensor; p is the mean stress; the positive variable λ represents the cumulative multiplier of the plastic strains; α, k and h are material parameters. In particular, the internal friction coefficient α and the initial cohesion k depend on the initial yield limits in tension and in compression, σ_{Yc} and σ_{Yt}, respectively, as follows

$$\alpha=\sqrt{3}\frac{\sigma_{Yc}-\sigma_{Yt}}{\sigma_{Yc}+\sigma_{Yt}}, \quad k=\frac{2}{\sqrt{3}}\frac{\sigma_{Yc}\sigma_{Yt}}{\sigma_{Yc}+\sigma_{Yt}} \tag{2}$$

Notably, the pressure–independent HHM model is recovered as α is assumed zero.
DP plasticity is coupled to a phenomenological model that describes the progressive degradation of the tensile material strength and stiffness leading to the complete material separation, typical of the brittle response observed in the experiment. In the considered approach, the onset of damage is met when the cumulative equivalent plastic deformation assumes a critical value. The damage evolution law is specified in terms of fracture energy, with linear dissipation. As irreversible strains increase, the yield limit and the entries of the elasticity tensor are decreased by the factor $(1 – D)$, where D is a measure of the accumulated damage. When $D = 1$ the material has lost its load-carrying capacity.
For the present application, some parameter has been fixed to the value reported in Table 2, assumed from literature data, in particular [6] and [7]. Others have been varied within a realistic range in order to evidence their role in controlling the coating integrity and the main damaging mechanisms.

Table 1. Mechanical properties of pipe steel

Young modulus E [GPa]	Poisson ratio ν [–]	yield limit σ_Y [MPa]	hardening exponent n [–]
180	0.3	400	0.08

Table 2. Mechanical properties of corrosion product layer

Young modulus E [GPa]	Poisson ratio ν [–]	compression yield σ_{Yc} [MPa]	hardening modulus h [MPa]	fracture strain ε_f [–]
150	0.3	400	60	0.01

Results

The preliminary numerical study reported herein concerns the simulation of conical (Rockwell) indentation tests performed on the bulk material coated by a single layer of corrosion products of about 15 μm thickness. The system response is assumed axisymmetric in order to reduce the computational burden required to understand the role of key mechanical parameters like the friction angle and the fracture energy, which distinguish the response of the coating in tension and compression. Results are compared in term of measurable quantities, defining the indentation curve and the geometry of residual imprint left on the specimen surface, as well as in terms of damage distribution.
The implemented FE models consider perfect interface between the bulk material and the corrosion layer, either smooth or with about 3 μm roughness height, as suggested by the micrographs reported in [4] and [8].

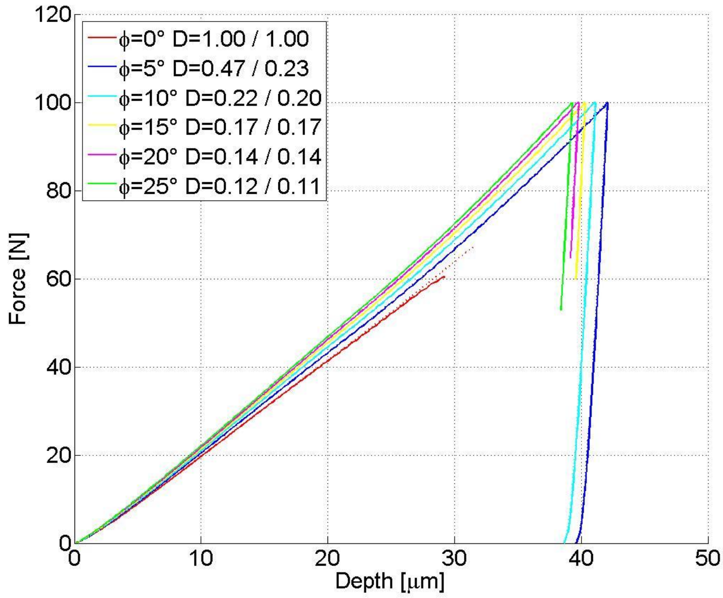

Fig. 2. Indentation curves for different friction angle and fracture energy fixed at the value $G_f = 1$ N/mm; continuous lines and the first *D* value refer to the computed system response for assumed smooth interface; the almost superimposed dotted lines and the *D* value after the forward slash correspond to the assumption of rough interface.

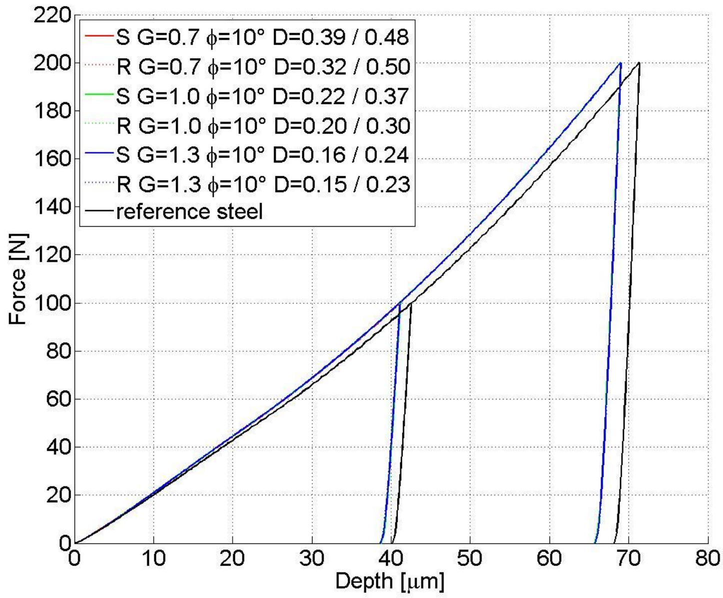

Fig. 3 Indentation curves obtained for $\Phi = 10°$ and for different fracture energy values compared with the response of the reference bulk material; the first *D* value refers to 100 N maximum load, the one after the forward slash to 200 N.

The results reported in Fig. 2 for a maximum 100 N load show the marked influence of the assumed friction coefficient on the overall stiffness of the system response: the increase of the α (= tan Φ) value reduces significantly the penetration depth. Notably, the accumulated damage (measured by the variable *D*) is also strongly affected by this material parameter and its value is maximized for the pressure independent simulation ($\alpha = \Phi = 0$). In this case, the material locally loses its load carrying capacity already for a maximum load of about 60 N. On the contrary, neither the indentation curves nor the amount of mechanical degradation experienced by the corrosion layer are

significantly influenced by the morphology of the interface between the coating and its substrate as $\Phi \geq 10°$.
The results presented in Fig. 3 shows that indentation curves are almost unaffected by the fracture energy, at least within the range $G_f = 0.7 \div 1.3$ N/mm assumed in the present investigation. However, larger G_f values facilitate convergence of the performed non–linear numerical analyses. The curves concerning the coated system are clearly distinguished from those relevant to the reference bulk material. The contribution of the corrosion layer reduces slightly the maximum penetration depth but has a marked effect on the piling-up height, which increases significantly (see Fig. 4).
The material is compacted under the indented tip while damage localizes in the piling up region as observed in the experiments; see [4]. A clear shearing mechanism appears in the case of a smooth separation between the coating and its substrate, as observed in [9], while the pattern is scattered by the interface roughness; see Fig. 5 and Fig. 6.

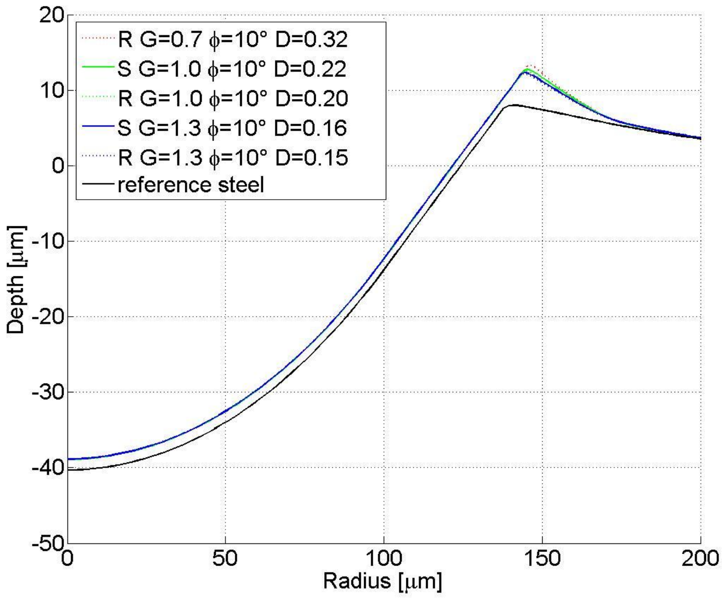

Fig. 4 Residual imprint obtained or at 100 N maximum load for $\Phi = 10°$ and for different fracture energy values; comparison with the response of the reference bulk material.

Closing Remarks

The numerical simulation of indentation tests performed on pipeline steel specimens coated by a single layer of corrosion products has been described in this contribution. The presented results were obtained by exploiting the modelling features available in a standard commercial code, which allowed to capture the principal characteristics of the observed system response, including the main damaging patterns. The model may be further refined in order to reproduce with improved realism the detachment of the corrosion layer from its substrate.
Tests performed on specimens with double corrosion coating evidenced the sharp propagation of discrete cracks along the interface of the two layers and inside the bulk material, reflected by jumps in the indentation curves. The interpretation of this experimental output will be the subject of future investigation.

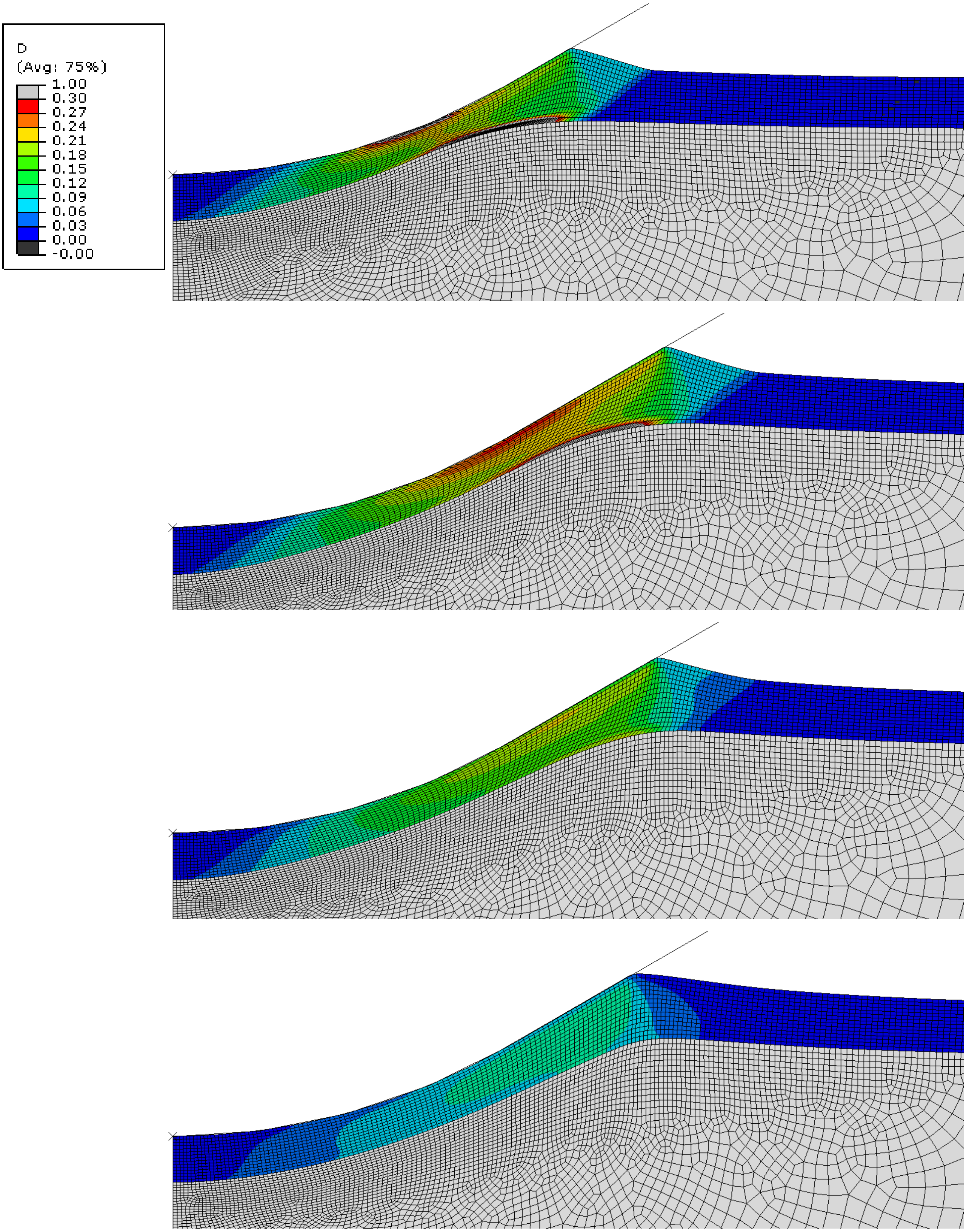

Fig. 5 Damage distribution for a smooth interface (Φ =0°, 5°, 10° and 25° from the top to the bottom; G_f = 1 N/mm) at failure (Φ =0°, see Fig. 2) or at 100 N maximum load.

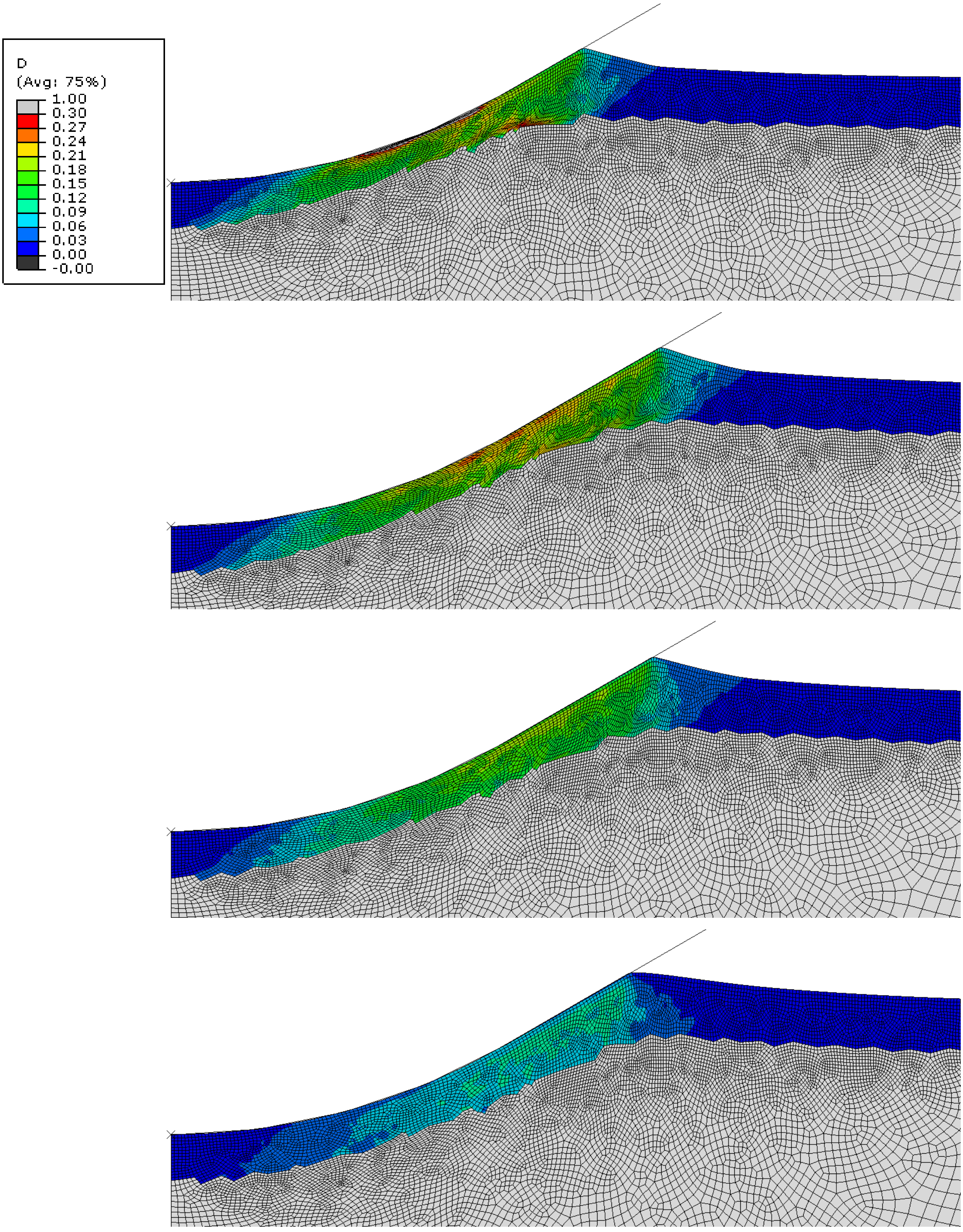

Fig. 6 Damage distribution in the case of a rough interface (Φ =0°, 5°, 10° and 25° from the top to the bottom; G_f = 1 N/mm) at failure (Φ =0°, see Fig. 2) or at 100 N maximum load.

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