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EXPERIMENTAL STUDY OF INELASTIC DEFORMATION AT THE MICRO SCALE IN CEMENTED GRANULAR MATERIALS: SOME RECENT RESULTS

Alessandro Tengattini[1], Itai Einav [1] and Gioacchino Viggiani[2]

[1] Particles and Grains Laboratory,
The University of Sydney, School of Civil Engineering, J05, NSW, 2006, Sydney, Australia
e-mail: alessandro.tengattini@sydney.edu.au

[2] Grenoble-INP/UJF-Grenoble 1/CNRS UMR 5521,
Laboratoire 3SR, 38041 Grenoble, France

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Abstract. A novel thermomechanical constitutive model for cemented granular materials has been recently introduced. An essential ingredient of the model is the use of measurable and micro-mechanics based internal variables describing the evolution of dominant inelastic processes. In this paper, discuss about the model ability to reproduce material behaviour at specimens scale starting from a few physically meaningful parameters. These parameters link the macroscopic mechanical behaviour to the statistically averaged evolution of the micro structure. However, to fully justify this statement and given the bottom-up hierarchy in the model development, it is also important to check the model's capability to capture the statistically averaged evolution of the micro structure embedded at its base. For that purpose we have used high resolution x-ray tomography to scan artificially cemented granular materials under a variety of loading conditions. X-Ray Tomography has been chosen for its capability to track inelastic processes in granular materials non destructively with a high spatial resolution (a few microns, in this study). The main objective of this work is to introduce the key features of the constitutive model and to report some recent results on the experimental quantification of the evolution of the microscopic internal variables in cemented granular materials.

1 INTRODUCTION

Cemented Granular Materials (CGMs) are a broad class of geomaterials in which densely packed particles are bridged by cement, which partially or completely fills the interstitial space [1, 2]. Within this broad definition fall a number of naturally occurring and artificial geomaterials (in Fig.1 are reported a few selected examples), in particular the focus of the proposed model are lightly to medium cemented granular materials (e.g. Fig.1, d-g). The similar micro-scale (grain-scale) texture implies that analogous micro-mechanisms lead the macroscopic behaviour: grain crushing, cement damage and fragment reorganisation [9, 10]. Existing models for CGMs ([11, 12, 13, 14]), which are often phenomenological adaptations of classic elastoplasticity for soil, have demonstrated their capability to reproduce the experimental data, yet the absence of direct links with the micro-scale processes seems to be the origin of the high number of parameters they require and of the lack of physical interpretation for some of them.

Recent technological developments give access to an increasing amount of experimental data about the evolution of the micro-scale properties, with an ever-improving spatial resolution. Also, the great advancements in computational power allows micro-mechanics based numerical methods such as DEM to get further insight into such phenomena. As our understanding of the physics involved at different scales broadens, new theories, capable of extracting and describing the crucial phenomena should follow, in order to practically implement this knowledge and lead further pertinent studies.

In the present paper the key elements of a micro-mechanics based model for CGMs are introduced, in §2, where particular focus is put over the choice of the internal variables. The practical feasibility of the experimental validation of model predictions at the micro-scale is studied in §3.

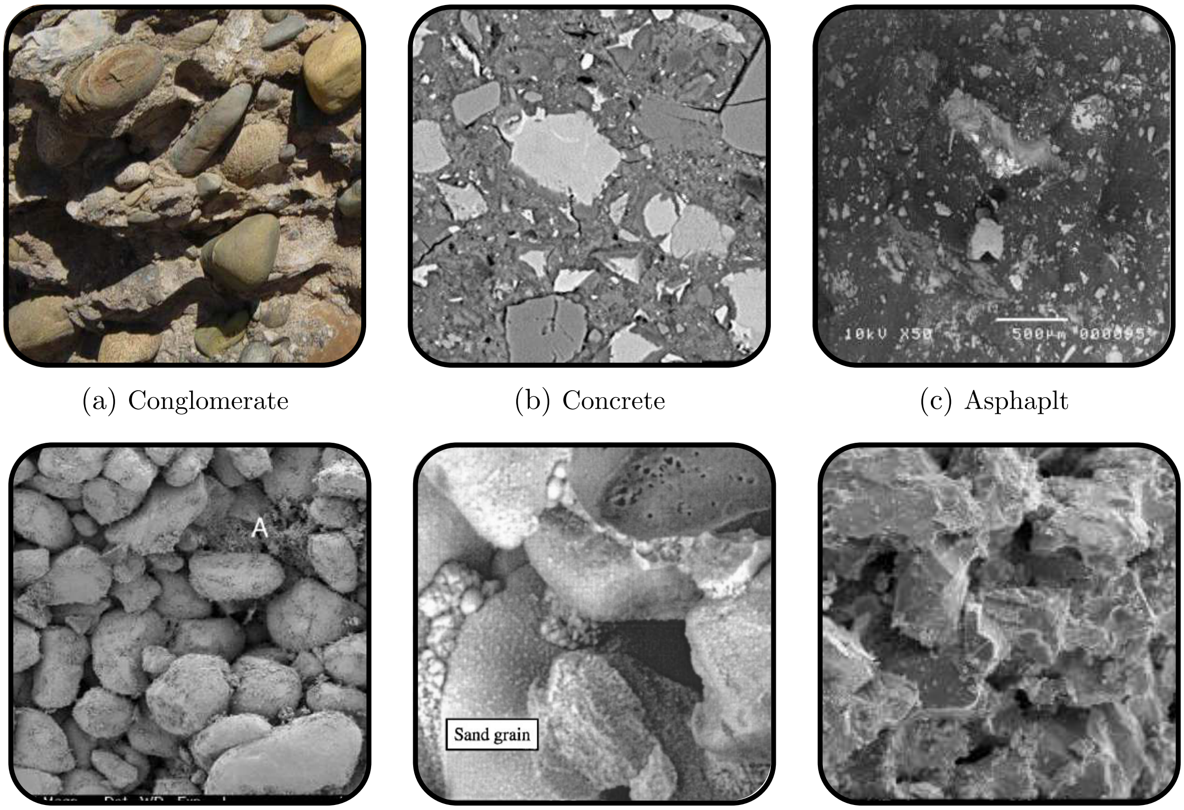
2 A CONSTITUTIVE MODEL FOR CEMENTED GRANULAR MATERIALS WITH MICRO-MECHANICS BASED INTERNAL VARIABLES

The main micro-mechanisms involved in the inelastic evolution of CGMs have long been studied in geomechanics [9, 10, 15] by means of electronic microscopy and acoustic emissions measurements. Such studies also evidenced the heterogeneity-dominated behaviour of the material at the micro-scale. The description of the physics involved should therefore be handled through statistical analyses. The recent availability of experimental tools (see §3) allowing full-field in-situ measurements of numerous properties and phenomena at the micro scale, have eventually given us access to the ideal experimental data required for a rigorous analysis of the physics involved.

This experimental state of the art calls for models aware of the micro-scale phenomena and capable of taking full advantage of the knowledge currently available or potentially achievable at the different scales. According to [16], it is in fact advised to implement, in the construction of constitutive models, *a transition between many stochastic processes on the microscale and a deterministic assessment of the macro response of a statistically homogeneous continuum.*

2.1 Internal variables

Particular attention has been given to the choice of internal variables which should ideally be identifiable, measurable and related to the dominant modes of irreversible rearrangements of the material microstucture. In fact *an internal variable inferred from*



(a) Conglomerate (b) Concrete (c) Asphaplt

(d) Saltwash Sandstone (e) Calcite cemented sand (f) Biocemented Sand

Figure 1: Examples of cemented granular materials: (a)-(c) heavily cemented granular materials [3, 4, 5], (d)-(f) lightly cemented granular materials [6, 7, 8].

the phenomenological evidence and selected to fit a particular stress strain-curve may provide a result that pleases the eye but seldom contributes to the understanding of the processes represented by the fitted curve. [17].

In the specific case of CGMs, the physics is driven by three main inelastic micro-scale processes [9, 10]: grain crushing, cement damage and fragment reorganization.

The description of the evolution of grain crushing has previously been successfully tackled taking into account the evolution of the grain size distribution. A scalar variable, *Breakage* is introduced as the area ratio in Fig.2a ([18, 19]). The advantages of such approach over the one proposed by the Continuum Damage Mechanics theory in the description of grain crushing in granular materials have been discussed in [18].

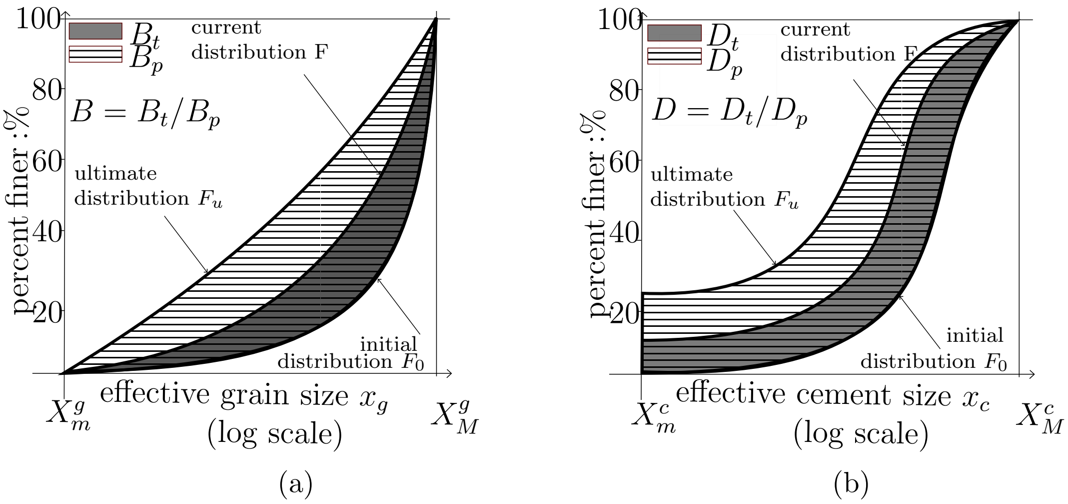


Figure 2: Scalar internal variables: a) Breakage (B), [18], b) Damage (D).

The behaviour of the cement phase is inherently different from that of the granular phase. The cement endows the system with tensile resistance, distributes the forces at the grain contacts, and enhances shear strength of the granular phase [20, 21, 22]. However, when cracks develop in the cement, the role of cement in the system is strongly redimensioned [23, 24]. Where the fragments of the crushed grains reorganise, entering in new force chains, the modest volume fraction of the cement in lightly cemented granular materials renders the mechanical contribution of its fragments to the force network negligible to a first approximation. It seems reasonable to describe this kind of behaviour as a progressive removal of the phase from the system, *i.e.* a damage-like approach.

Citing (again) [16], *the statistical characterization must emphasize the universal aspects and simultaneously blur away the inconsequential details*, depending on the *purpose of the analysis and the required resolution of the model.* The proposed solution for CGMs is to take into account only the cement that is effectively working. When a cement bridge is partially cracked, we can in principle substitute it with another, with a smaller sectional area, but made of undamaged material with the same contribution to the system, as sketched in Fig.3. It is then possible to define an effective cement size distribution through which we can describe the evolution of damage. Analogously to the *Breakage* (B) variable it is possible to define a scalar *Damage* variable (D) as the area ratio in Fig.2b. While the conceptual measurability of this internal variable seems legitimate, the possibility of a direct experimental assessment is discussed in § 3.

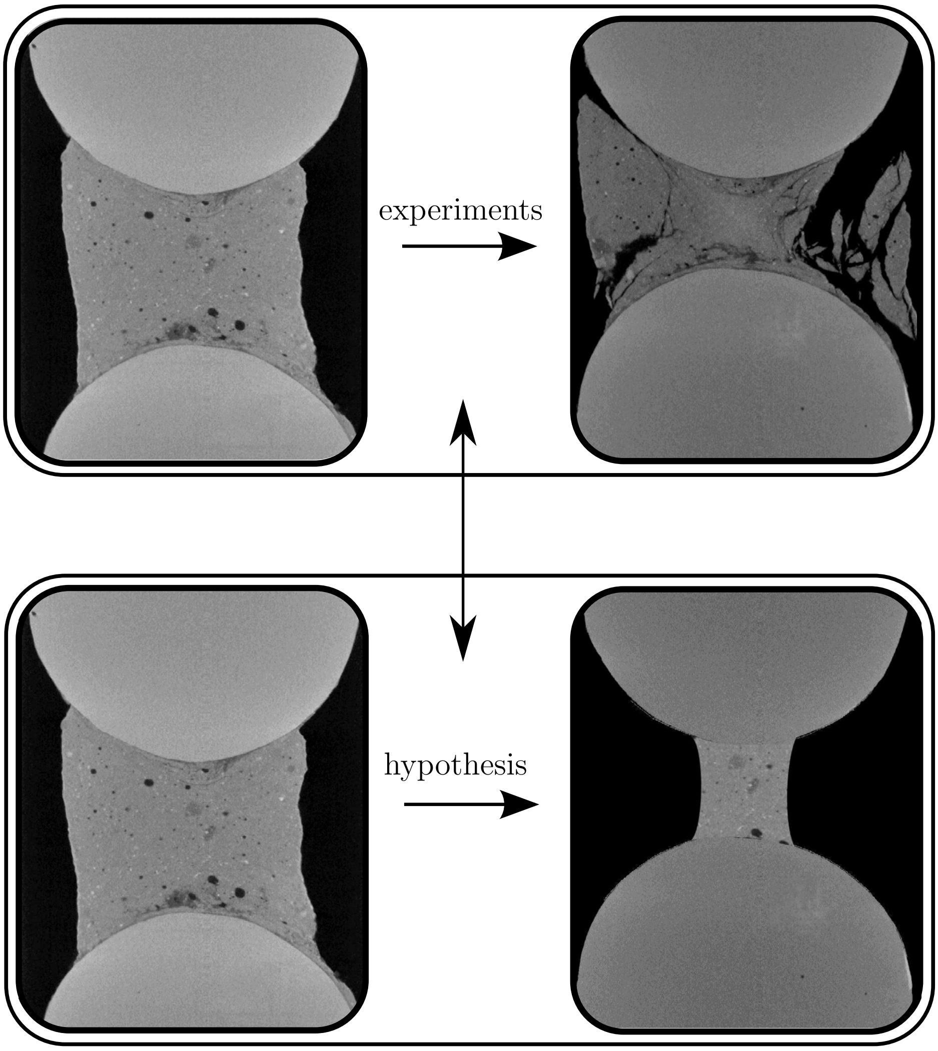


Figure 3: Sketch of the mechanical equivalence assumption. Top raw: damage as observed experimentally. Bottom raw: way in which damage is taken into account in the model

The last inelastic microscopic process considered, fragment reorganisation, can be effectively characterised by the plastic strain, which, describing the unrecoverable macroscopic strain, lends itself as an natural measure for this phenomenon.

2.2 The model at a glance

Beyond the possibility of having observable (at least in principle) descriptors of the micro-scale texture, the use of physically meaningful internal variables allows a rational connection between them, the evolution of the elastically stored energy and the energy dissipation.

As shown in [18], in fact, it is possible to describe, through statistical homogenization over a Representative Elementary Volume, the elastically stored energy in a granular material describing how the Helmholtz free energy is distributed according to the grain size. It is then possible to deduce the evolution of the elastically stored energy as grain crushing proceeds and to connect its loss to the increment of dissipation. Imposing the first two laws of thermodynamics, it is eventually possible to deduce the full constitutive model.

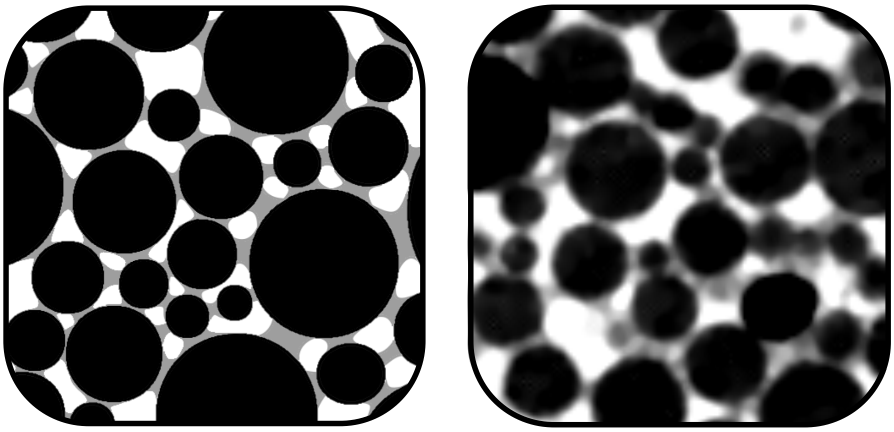
In the proposed model an homologous approach is adopted to describe the energy stored in the grain phase and an analogous description has been made for the cement phase. While the full derivation of the model can be retrieved in [25, 26] the final constitutive equations can be found in [27].

3 MODEL VALIDATION

As detailed in [25, 26], the proposed model requires only 8 parameters and 3 geometrical indexes, each having a precise physical interpretation, to be compared with the 13 or more parameters of well-established models [12, 13]. While the capability of the model to reproduce the mechanical behaviour of some selected CGMs is analysed in [26], both at the material point and at the structural (specimen) scale, the aim of this paper is to to explore the intriguing possibility to also verify the model predictions regarding the evolution of the microstructure.

Recently, a number of experimental tools have been refined, allowing access to full-field measures of disparate mechanical informations at the micro (grain) scale in CGMs [10, 28]. A common element in the different technologies seems to be the trade off between the spatial resolution and the size of the considered specimen, that should be bigger than the Representative Elementary Volume to be interesting from a statistical viewpoint. The study of the acoustic emissions gives access to the distribution of the inelastic events of elastic energy release [29, 30], suggesting the fascinating possibility of measuring at least some of the energy dissipated in the system directly. Also, the use of ultrasonic tomography has lately shown its capability to get to full field measures of the elastic properties [31].

In this work we focus on high resolution x-ray Computed Tomography (CT), which has lately shown the potential of the reconstructed 3D map of absorption in the analysis of grain scale processes. In particular, focusing on the phenomena of interest for CGMs, a way to estimate grain crushing has recently been proposed in CGMs [32] in cases where the spatial resolution is not sufficient to discern the single grain fragments. While the inelastic rearrangements have often been studied at the mesoscale (ignoring the discrete nature of the parts), recent developments have shown the possibility to follow the kinematics of individual elements [33]. To the best of the authors' knowledge the process which has been less extensively studied, at least in terms of full field experimental measures, is the evolution of damage. With the aim of analysing cement fragmentation alone, a model material has been designed to prevent other inelastic phenomena such as grain crushing. Also, since a simple geometry of the granular phase allows to take advantage of the grain topology for image post-processing, spheres have been selected. The material proposed is calcite-cemented glass ballottini, with a D50 of about 300μm. Fig. 4 shows the similitude between the conceptual scheme behind the model and the proposed material.



(a) (b)

Figure 4: Idealized Cemented Granular Material: a) schematic section of CGMs,representing a concept embedded in proposed model b) X-Ray image of Calcite cemented Glass Ballottini, model material well fitting the conceptual scheme.

The tested specimens are cylindrical, with a diameter of 11 mm and a height of 22mm, for a total of more than 50k grains. The degree of cementation is, to a first approximation, 10% in terms of volume. In Fig.5, a cross section of the 3D reconstruction coming from the x-ray CT (about 10 μm resolution) of the specimen before and after a triaxial compression test are shown. It can be readily seen how the material undergoes cement damage without developing grain crushing. While the qualitative evolution of the damage along the test can be easily observed, accessing the quantitative measurements necessary to study the evolution of the cement damage requires a non-trivial analysis of the images. To assign the voxels to one of the phases, an elementary approach is to classify them based on their grey value: the x-ray absorption of calcite lies in between the one of the grains and the one of the voids. A shortcoming of the method is the partial volume effect ([34]): voxels lying on the surface of the grains and partially representative of two or more phases will have a grey value similar to the one of the cement. To deal with it, a dilation can be applied to the grain phase to include in this category the voxels on the surface of the grains and suffering of the partial volume effect. Fig.6 shows the application of the method for the test in Fig.5.

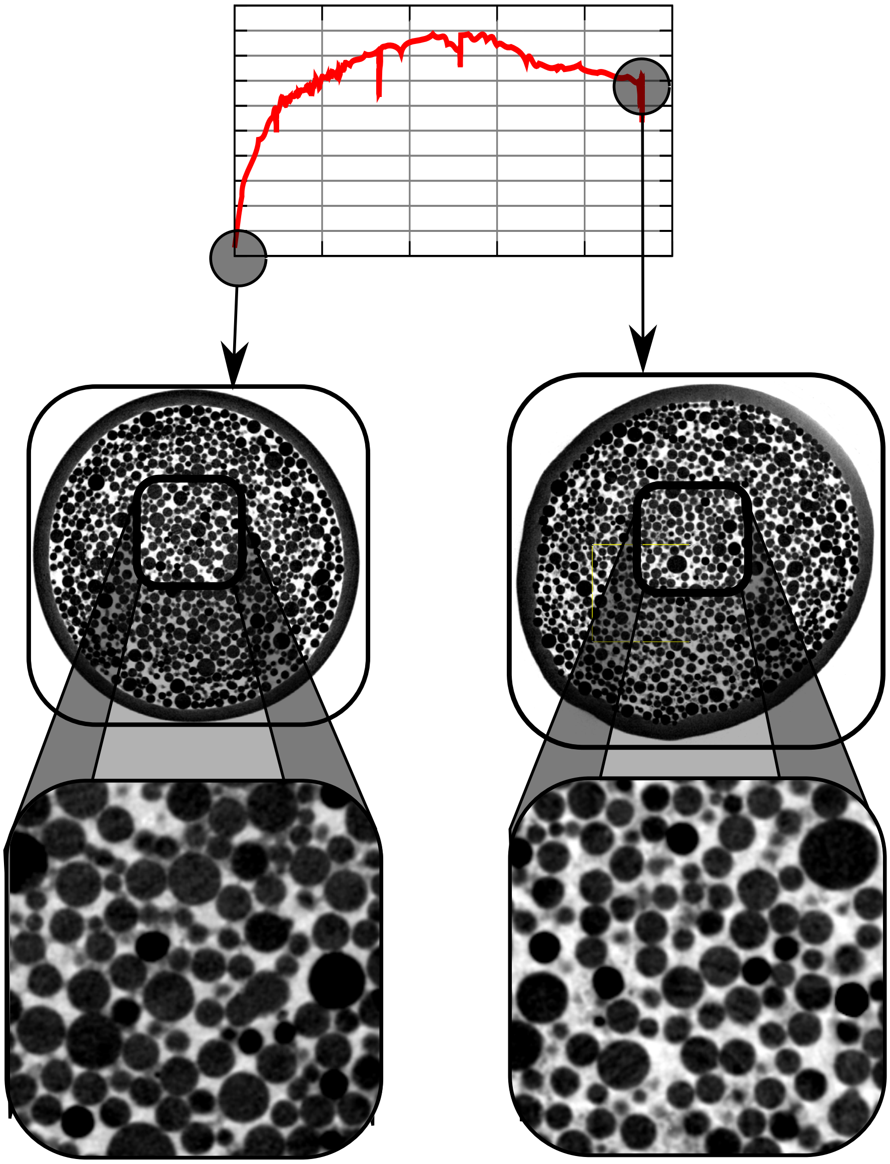


Figure 5: Evolution of cement damage, seen through the loss of the bond's grey scale

To apply this technique it is necessary to define the boundaries of grey-scale values of the different classes. An estimate of the total volume of the grains and cement phase, deduced from the total mass of the sample and the proportions used in the sample construction, can be used to impose the number of voxels in each category for the initial stage.

As the cement damage proceeds the cement fragments detach from the cement bridges and the remains, now below the spatial resolution, will not be any more detectable. In the example reported in Fig.6 the degree of cementation (V_{cem}/V_{tot}) goes from 10 % to 4% (accompained also by an increase of porosity from 14% to 27%, in the picture). Although the values of the degree of cementation might be argued due to the simplicity of the technique, their increment seems a reliable information. The process required to get more robust and objective measures of the cement size distribution is yet to be refined. Nonetheless, the amount of voxels per cement bridge (few hundreds) seems sufficient, at least in principle, to describe the evolution of the cement damage.

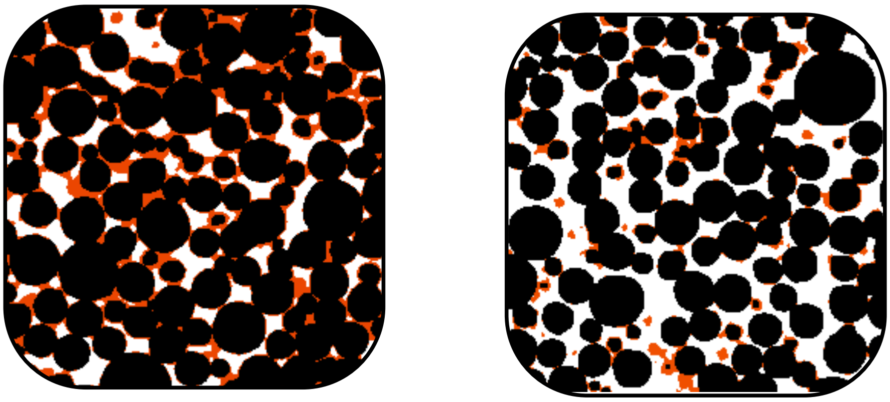


Figure 6: Evolution of cement damage

4 CONCLUSIONS

In the present paper the fundamental hypotheses of a novel micro-mechanics based constitutive model for Cemented Granular Materials are discussed. The model takes into account the main experimentally observed inelastic processes. Particular attention is dedicated to the definition of the internal variables that allow to track the evolution of the material micro-structure and connect it, through statistical homogenization, to the macro-scale response. This model tries to take advantage of the increasing amount of quantitative experimental data regarding the processes occurring at the micro-scale. While the conceptual measurably and the mechanical relevance of these variables seems legitimate, the intriguing possibility of gathering experimental full-field measures of these internal variables is discussed. In particular the practical feasibility of the isolation of damage is here shown in an on-purpose designed model material. The main issues that have to be faced in the quantitative analysis of the process are also outlined. Although the process is certainly not trivial, the possibility of verifying the model's predictions both at the macro and micro scales have important repercussions both on the validation of the proposed approach but also, perspectively, on the study of other important micro-scale properties such as the permeability.

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