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Effective elastic moduli of a heterogeneous oolitic rock containing 3-D irregularly shaped pores

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ABSTRACT

We have characterized the microstructure of a heterogeneous oolitic rock (limestone from Lavoux, France) with X-ray nano-computed tomography. This rock comprises an assemblage of porous grains (oolites), irregularly shaped three-dimensional pores, and inter-oolitic crystals (cement). To model the effect of this microstructure on the macroscopic behavior of the rock, we approximate the porous oolites by spheres, and the irregularly shaped pores by ellipsoids. This approximation is performed based on the principal component analysis PCA, which provides the geometrical properties such as length of semi-axes and orientation of resulting ellipsoids. The sphericity of the approximated oolites was calculated and the value close to 1 allows us to consider oolites as spheres. To verify the approximation in the case of pores, we evaluated the contribution of these irregularly shaped three-dimensional pores to the overall elastic properties. Thus, compliance contribution tensors for 3D irregular pores and their ellipsoidal approximations are calculated using the finite element method. These tensors were compared to the compliance tensors for ellipsoids obtained using analytical solutions based on Eshelby's theory and a relative error is estimated to evaluate the accuracy of the approximation. This error produces a maximum discrepancy of 4.5% between the two solutions respectively of pores and ellipsoids which verifies the proposed approximation procedure based on principal component analysis. The FEA numerical method is verified by comparing the numerical solution of compliance contribution tensors of the ellipsoids to the known analytical solution of these same shapes based on Eshelby's theory. The difference between these two solutions does not exceed 3%. Compliance contribution tensors are finally used to compare effective elastic parameters of a material containing irregular pores via the Maxwell homogenization scheme. These elastic parameters (bulk modulus and shear coefficient) coincide with a maximum deviation of 5% with ones for a material with ellipsoidal pores.

1. Introduction

In this paper, we propose a computational procedure to characterize the geometry of the microstructure and calculate effective elastic properties of a heterogeneous porous rock, an oolitic limestone from Lavoux (France). This rock was chosen due to its relative simple microstructure organization and it is composed by an assemblage of porous grains (oolites), non-ellipsoidal irregularly shaped pores and inter-granular crystals (cement).[1–5] The microstructure of the material was observed using 3D X-ray nano-computed tomography and scanning electron microscopy SEM. We analyze then the X-Ray images using VGStudio MAX, an image processing software developed by Volume Graphics (www.volumegraphics.com), in order to distinguish the different components of the material. We are interested particularly in the characterization of two main components of this rock: oolites and pores while inter-granular crystals (cement) were considered as a homogeneous matrix, where the pores are embedded, in the mechanical problem evaluation because it is composed of 100% of calcite. The simplified model was considered within the framework of the Maxwell homogenization model which will be used in this study. The advantage of this simplified model is reflected in its simplicity in the application of the corresponding homogenization model. As in 6, this simplified model consists in approximating porous oolites by spheres, and irregularly shaped pores by ellipsoids with randomly distributed orientation. The verification of the accuracy of the pores approximation by ellipsoids will be done through the determination of the effective elastic properties. For this goal, we evaluate compliance contribution tensors that represent the extra strain due to the presence of a single inhomogeneity in a matrix subjected to a uniform stress field. The novelty of this work consists to take into account the irregularly shaped 3D pores of a rock which are modeled by ellipsoids thanks to the principal component analysis PCA. Available results

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for 3D irregular pore shapes are limited in the literature. For 2D shapes, one can refer to classical schemes based on Eshelby solution for ellipsoidal shapes[7] to evaluate analytically the contribution of pores to effective elastic properties. For instance, 2D pores of ellipsoidal shape were studied by 8 and compliance tensors for these pores were calculated. Numerical solution for 2D shapes can be found in 8–10. For 3D inhomogeneities, analytical solutions for non-ellipsoidal shapes were presented in 11–13. In our case, analytical solution is not relevant due to the high irregularity of pores, so numerical methods such as finite element analysis (FEA) was used. In the context of irregularly shaped cracks, one can refer to 14,15. Compliance contribution tensors for a material containing concave pores were determined by 16,17. using semi-analytical method based on finite element analysis where several pores were studied and characterized by a shape factor in order to evaluate effective elastic properties. In addition, compliance contribution tensors for 3D irregular shapes pores typical for carbon-carbon composites were evaluated by 18 by finite element analysis. The authors used PCA method to approximate the irregularly shaped pores by ellipsoids. In the same way, FEA was used in 19 to compute linear elastic properties from micrographic images. The method presented in this paper to determine contribution tensors, combines analytical and numerical techniques. Hence, contribution tensors for irregularly shaped pores are evaluated numerically while those of the ellipsoids are evaluated using both approaches. The FEA procedure for the evaluation of the contribution tensors of irregularly shaped pores and their corresponding ellipsoids is presented after considering isolated pores embedded in a homogeneous matrix. After that, the calculation of contribution tensors of the approximated ellipsoids using Eshelby solutions is described and a relative error due to this approximation is estimated.

Calculated tensors are used then to evaluate effective elastic properties (bulk modulus and shear coefficient). The dependencies of these elastic parameters (bulk modulus and shear coefficient) on the volume fraction are presented after considering Maxwell homogenization scheme. The results obtained on irregular pores are finally compared to those obtained on ellipsoidal pores to evaluate the effect of the approximation on effective elastic properties.

2. Microscopic observations and microstructural characterization of Lavoux limestone

2.1. Description of the microstructure of a reference porous oolitic limestone

Mineralogical and textural analysis of Lavoux limestone using scanning electron microscopy (SEM) and optical microscopy showed that it is made of oolites affected by early diagenesis marked by the presence of large syntaxial calcite crystals around residues of echinoderms. The size of the oolites, which are made up of concentric layers of microcalcite and bound by calcite cement, varies between 100 and 1000 μm. Three main types of calcite crystals can be found: large syntaxial crystals of several hundreds of micrometers, small equant calcites with an average size of 10 μm precipitated around the oolites in vadose conditions and microcalcites with an average size of 3 μm in the core of the oolites.[20] It is possible, however, that zones of micrite (crystals of 1–4 μm) ensure the cohesion of the grains[21,22] (Fig. 1).

The chemical characterization performed on Lavoux limestone by Energy Dispersive X-ray Spectroscopy method (EDS) under SEM has shown that the material is mainly composed of calcite and contains a very small fraction of clays and dolomite.[23] Indeed, the nominal composition in atomic percentage measured using EDS is: O (75.3%), Mg (0.16%), AL (0.5%), Si (0.29%), Ca (23.42%), Fe (0.32%).[2] Therefore, one can consider this material as almost a mono-mineral limestone composed totally of calcite so that intergranular calcite cement is considered homogeneous.[3]

To characterize the microstructure of Lavoux limestone, we used 3D X-Ray nano-computed tomography. Tomographic data were obtained using a synchroton Phoenix nanotomogaph at the GeoRessources Laboratory (University of Lorraine, France). The scanned sample has a size of 5 mm of diameter and the resolution of the scan was set to 5 μm. Raw gray scale reconstructed tomographic images contained data of all the constituents of the material. A thresholding algorithm was applied to convert these data into series of gray and black images where the black represents the pores and the gray represents the solid constituents. To obtain the pore structure for future micromechanical modeling, a representative elementary volume (REV) of 1 mm^3 volume was selected from the binary database. The analysis of the REV using image processing software showed that the limestone consists of three main components (Fig. 3): oolites having more or less spherical shape (Fig. 3a), interoolitic pores having irregular shapes, and interoolitic crystals (Fig. 3c). By applying a segmentation algorithm based on grayscale values, we obtained the porous network of the REV (Fig. 3b). The calculated porosity is equal to 6.9% and it represents the volume of inter-oolitic pores (meso and macro pores) over the total volume. Micropores inside oolites are not taken into account because they are not accessible due to the limitation of the resolution (5 μm) of X-ray tomography images. This porosity value is compared to the value obtained from mercury intrusion porosimetry. Grgic[2] applied this technique on Lavoux limestone and obtained a spectrum containing a bimodal distribution of pore access size corresponding to two kinds of pores (Fig. 2): intra-oolitic pores ($0.002\ \mu m < r < 2\ \mu m$) and inter-oolitic pores ($2\ \mu m < r < 50\ \mu m$), where r represents the entrance radius of pores. By considering entrance pore radius larger than 5 μm, which corresponds to the tomography resolution, mercury porosity is equal to 7%, which is very close to 6.9%.

2.2. Approximation of irregularly shaped elements by ellipsoids

2.2.1. Pores

In this paper, we are interested in the characterization of the shape of two important components of the material which are pores and oolites. Thus, from a micro-mechanical point of view, the microstructure of the investigated material is characterized by grains (oolites) surrounded by a matrix and both of them are porous materials. In general, a regular shape can be specified by geometrical parameters in relation with size. For example, the diameter of a sphere, the aspect ratio and the length of one axis of a prolate or oblate spheroid in two dimensions or the semi-axis lengths of an ellipsoid in three dimensions. In other words, the quantification of such types of shapes is based on the analytical relation between their dimensions and their geometrical properties like volume and surface.[24] The characterization of irregular shapes requires an important number of parameters. For example, 25 presented 10 parameters to quantify an irregular shape and[26] listed even more. The dimensions of 3D irregular shapes are usually defined using equivalent shape method. To construct the equivalent regular shape, one can follow these steps: One or more geometrical parameters are selected (volume, surface, inertia moments, etc.). These parameters are evaluated by some means for the irregular particle, and finally a regular shape is selected, and its dimensions are determined by equating the geometrical properties of the irregular shape to the analytically known geometrical properties of the regular shape and solving the dimensions of the regular shape.[24]

Approximating 3D irregularly shaped pores (or other components) by ellipsoidal shapes is very common practice in evaluating effective properties. Indeed, ellipsoids have the property of uniform eigenstrain under remotely applied loading, so that the analytical solutions for strain and stresses can be used.[7,27] The approximation of irregularly shaped pores by ellipsoids can be based on several parameters depending on the application domain. In fact, two issues may arise: the first is the choice of the best approximation of pores shape by ellipsoid (orientations and lengths of the principal axes) and the second is the accuracy of the selected approximation.[18] In this paper, we focus on mechanical applications that require the conservation of inertia moments of initial pores shape. Thus, we chose PCA method to