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Microstructural changes in clays generated by compression explored by means of SEM and Image Processing

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Abstract

A study on the microstructure of an illitic marine clay is carried out through a thorough investigation of the clay origin, composition and current microstructure. The clay fabric is investigated by means of scanning electron microscopy (SEM) and statistically analysed by means of image processing. The nature and strength of the clay bonding are probed by means of direct chemical micro-analyses and on purpose strain paths. The same investigation is carried out both on the natural and on the reconstituted clay, at the initial state and after one-dimensional compression to medium and large pressures.

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Keywords: aggregates; clays; fabric/microstructure; laboratory; microscopy

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1. Introduction

The hydro-mechanical behavior of clays is most often modelled according to the principles of continuum mechanics in the theoretical framework of elasto-plasticity. Several constitutive models are available to represent the response of the clay recorded at the scale of the element volume (macro-response in the following) as it was exhibited by an elasto-plastic continuum, whether the clay is reconstituted in the laboratory, or is natural [1-4].

Given the complexity of the chemo-physical processes that occur at the micro-scale [5-8] and control the clay macro-response, its prediction through the modeling of such processes is a major challenge. To make the knowledge about the processes that, at the micro-scale, are background of the clay macro-response, it is rational to investigate

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the micro-structural features of the clay when at given stress-strain states and their evolution along given stress-strain paths (i.e. while the clay exhibits given macro-responses). This investigation can nowadays make use of the greatly advanced technical procedures that, to date, can explore materials at the micro-scale. So, for example, recording the variations in the microstructural features of the clay that take place with plastic straining along given loading paths, would allow to assess the genesis of the hardening behavior being recorded at the macro-scale and would provide evidence of the physical processes whose results are simulated by the hardening law. At the same time, the data acquired at the micro-scale and their phenomenological interpretation may configure the ground for advancements in the micro-mechanical modeling of clays.

The present paper reports some of the results of a long lasting investigation of the processes that, at the micro-scale, occur while the clay exhibits given macro-responses. The investigation aim is at characterizing the micro-scale physical features that determine given macro-responses, in order to relate different facets of macro-behavior, as well as the corresponding modeling algorithms, to the nature and microstructural features of the clay. In this way, classes of clays, distinguished on the basis of the micro-structural features, could be connected to classes of macro-responses and, hence, to constitutive laws and parameter values. In the research here of reference, constitutive laws framed in the theory of elasto-plasticity have been considered, to start with.

The availability of a correspondence between clay typologies, constitutive models and parameter values should prompt a wider use of the constitutive models in practice, since it would support the engineer in the selection of the model and of the parameter values most appropriate in the design, given the clay response he has to predict.

The research procedure has entailed the investigation of the microstructure of the clay for different stress-strain states. In particular, natural and reconstituted clays have been investigated, in the logic of comparing clays of identical composition, but different structure as result of the differences in deposition conditions and stress-strain history [9-11]. To this purpose, laboratory element tests on both natural clay and reconstituted clay specimens have been carried out and the tested specimens have been subjected to microstructural analyses, thereafter. The latter have been carried out by means of scanning electron microscopy (SEM), image processing [12] and swelling tests.

The image analyses aim at a quantitative assessment of the fabric orientation and consist in the digital processing of the SEM pictures. This processing results in the thinning of the brightest pixels of the SEM picture, from which the directions of the particle contours are derived. Through statistical analyses of the oriented segments that result from the thinning procedure, that are considered to correspond to the laminae contours, or edges, a direction histogram is determined. The degree of particle orientation is derived from the histogram and delivered as ‘index of fabric orientation’, L [12, 5]. The value of L can range from 0, for randomly oriented particles, to 1, for perfectly oriented fabric; for $L>0.21$, the fabric is considered well oriented. At the same time, the bonding nature is characterized with chemical micro-probing in the SEM, while the bonding strength is characterized indirectly through swelling tests [e.g. 13-16].

2. Differences in state and state boundary surface of clays of different microstructure

To start with, it is worth recalling the definition of clay microstructure. This is the combination of the geometric arrangement of particles, defined as fabric, and all the inter-particle forces that are not of mechanical nature, but are rather chemical, electrostatic, or electro-magnetic, which determine the clay bonding.

The clay microstructure controls the state of the material, expressed in terms of void ratio, e, and corresponding vertical effective stress, σ'_v, when in geostatic conditions, and is, in turn, determined by the clay composition, the deposition conditions, the clay loading history and other geological processes that may occur under burial [7,8,17]. For a given composition, deposition conditions and one-dimensional loading in situ, the clay state evolves under burial along the so-called sedimentation compression curve (SCC, [3]), controlled by the sedimentation structure of the clay [9,11,18]. If diagenesis occurs, the structure may develop further, in a post-sedimentation structure; for example diagenesis may generate a strengthening of bonding, in which case an increase in strength and yield stresses are recorded [18,19].

The research programme has first addressed the detection of the differences in microstructure that a clay of given composition can acquire when sedimenting and consolidating in situ in its geological history, or otherwise in the laboratory, with normal-consolidation after reconstitution [9]. Since natural and reconstituted clay specimens, experiencing such differences in sedimenting and consolidation history, have been found to exhibit different shear strength properties and compression gross yield pressures [9,18], the differences in microstructure being explored are to be considered the cause of such differences in macro-response.

In particular, irrespective of composition, all the clays reconstituted in the laboratory are found to possess a microstructure that is the weakest possible and is not sensitive to disturbance, i.e. its strength properties do not impoverish with straining [9,18]. Critical state soil mechanics [20,21] has been developed based upon the macro-behavior of clays possessing this structure and, since then, Cam-Clay like constitutive models, with an isotropic volumetric hardening, have been developed to simulate the elasto-plastic behavior of these clays [1,4]. Natural clays may differ in structure from reconstituted clays, this difference being implemented as additional internal variable influencing the clay hardening law with compression and the clay response to shearing.

If normalized for composition [9,11], the sedimentation compression curves of reconstituted clays are all represented by a single Intrinsic Compression Line (ICL, [9,18,19], Fig.1a). Conversely, the compression states of natural clays normalized for composition can lie to the right of the ICL, if the natural clay structure is stronger than that of the same clay when reconstituted (Fig.1a). Cotecchia & Chandler [19] suggested to relate the macro-effects of such differences in microstructure to a scalar parameter, called stress sensitivity S_σ, defined as the ratio of the yield pressure of the natural clay under 1D compression and the equivalent pressure along the ICL. Figure1a shows the compression behavior of different natural stiff clays normalized for composition, which possess low values of void index I_v at high vertical effective pressures, compared to the state of a sensitive soft clay, Bothkennar clay, that locates at high I_v and low vertical effective pressures, compared with the ICL. The soft and the stiff clays in the figure possess very different microstructures, which in turn are all different from that of the same clay when reconstituted and compressing along the ICL. From the analysis of several data, the increase in stress sensitivity S_σ of clays, i.e. of the distance of the compression yield state from the ICL in the I_v - σ'_v plot (Fig. 1a), has been found to correspond to the increase in size of the whole state boundary surface (SBS) of the clay, with respect to the SBS of the same clay when reconstituted [18]. This positive hardening is due to a strengthening of structure that may be recorded for natural clays due to the geological processes, that is not applying to the reconstituted clay, and is gradually lost over compression post-gross yield, with a consequent negative effect on the clay hardening [2,3,18].

In the following, the differences in microstructure between a natural clay that has experienced important diagenesis in its geological history and exhibits S_σ = 3, and the same clay when reconstituted in the laboratory are discussed, when the clays are either in the initial state, or after compression post-gross yield. The latter determines different hardening for the two materials; hence the microstructural changes causing the different hardening laws are explored.

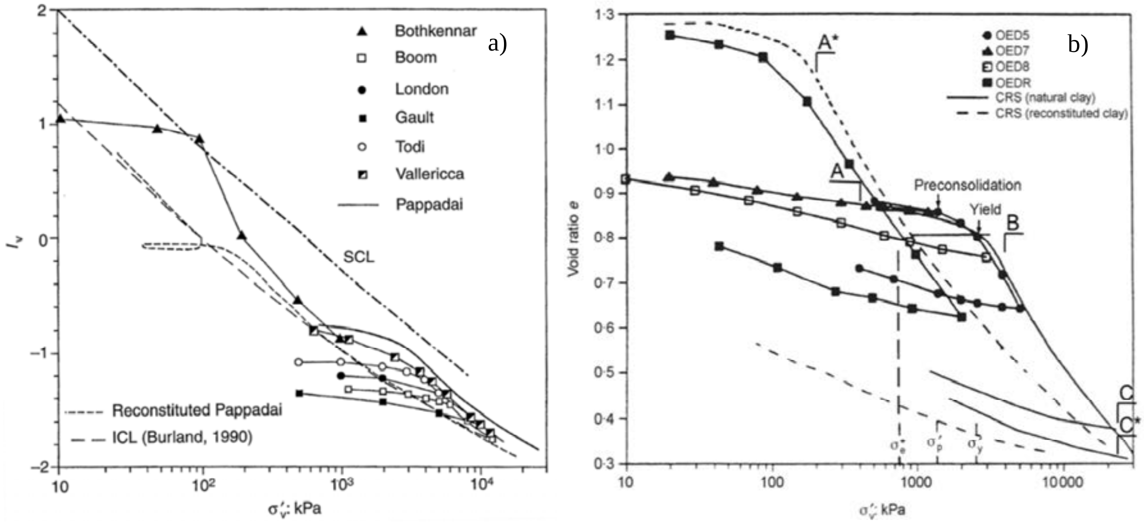

Fig. 1. One-dimensional compression behavior a) of natural and reconstituted clays normalized for composition and b) of natural and reconstituted Pappadai clay subjected to different oedometer (OED) and constant rate of strain (CRS) tests (adapted from [18, 19]).

3. Microstructural features in the background of the observed differences in macro-behaviour

3.1. Tested material: composition and 1D compression states of the natural and of the reconstituted clay

The tested material is a clay of marine origin, the Pappadai clay, mainly illitic, of high plasticity, medium activity and high carbonate content. The natural samples were deposited in the Montemesola basin (Southern Italy) in the mid-Pleistocene. According to the paleontological analyses [14], deposition occurred in protected highly still water, allowing for a reducing environment. The stillness of the water is confirmed by the lamination of the clay; in particular, strata of coccoliths, i.e. calcareous nanofossils, are detected along the lamination planes. The analysis of mineralogical profiles in the deposit [14] allows to recognize typical effects of diagenesis, resulting in a decrease in the proportion of smectites and an increase in the proportion of non-swelling minerals with depth. Therefore, additional bonding is likely to have developed in the natural clay due to diagenesis under burial. The clay was later overconsolidated due to erosion, the expected geological preconsolidation pressure being σ'_p 1300 kPa and the overconsolidation ratio OCR=3.2.

Block samples of the natural clay were taken from about 25 m depth down a shaft [15,19] and one-dimensionally compressed in the laboratory (Fig.1b). A gross yield pressure of 2600 kPa has been measured in the compression tests, with a yield stress ratio YSR=6.4 [9], that is twice the OCR. This result gives evidence to the strengthening suffered by the clay microstructure under burial, as result of the diagenesis [18,19]. Also the reconstituted Pappadai clay has been one-dimensionally compressed in the laboratory. The measured ICL is shown in Figure1b to plot to the left of the gross yield state and of the post-gross yield compression curve of the natural clay. The maximum value of the compression index of the reconstituted clay, C_c*, is about 0.47, lower than that of the natural clay, C_c, which is about 0.56. The natural clay is found to exhibit S_σ=3, that synthesises the difference in microstructural strength between the undisturbed natural and the reconstituted Pappadai clay. With compression to very high pressures the natural and the reconstituted states get closer, but they do not converge along the same curve (Fig.1b); evidently their microstructures keep being different over the wide range of pressures being crossed. Changes in bonding strength can be assessed through the analysis of the changes in swell sensitivity, that is the ratio of the swelling index of the reconstituted clay, C_s*, to that of the natural clay, C_s [16]. Swell sensitivity equals 2.5 for the undisturbed natural clay, indicating that the swelling of the natural clay pre-yield is constrained by bonding; however, C_s*/C_s reduces to unity post gross-yield, suggesting that bonding weakens with compression and is lost quite immediately over gross yield.

3.2. Microstructure of the undisturbed natural clay and of the reconstituted clay

The microstructure on vertical fractures of both the natural Pappadai clay and of the same when reconstituted has been analysed by means of SEM. The samples were subjected to freeze-drying and gold-coating. Microscopic observations can capture the clay fabric at different levels of magnification: a segment of 1 cm length on the micrograph corresponds to hundreds, tens and units of μm for the large, medium and small magnification, respectively. The reconstituted clay fabric at point A* (Fig.1b), for medium magnification, is shown in Figure 2a. From a qualitative point of view, the fabric appears rather packed and complex: both stacks, i.e. strata of complete preferred orientation [7], and areas of randomly oriented fabric can be recognised, indicating a non-ubiquitous full orientation. The index of fabric orientation, L=0.27, confirms that the fabric is on average well oriented. Fig. 2b shows the fabric of the natural undisturbed Pappadai clay (point A, Fig.1b) at medium magnification. The clay fabric appears as of a highly compressed ‘bookhouse’ type, in which both very dense stacks and randomly oriented areas can be identified, and overall has a medium orientation. The observed value of L confirms the qualitative interpretation. Chemical micro-probing in the SEM has also provided indication of the presence of an amorphous calcite film [14] on all the particles, which seems to be binding the particles. This film represents the effect of diagenesis and, as such, the factor increasing the significant bonding present in the clay, also manifested by the high swelling index quoted above. Noticeably, the higher magnification SEM pictures of portions of either the natural, or the reconstituted clay, which are shown in Figure 3, show a much less oriented fabric, quantified by a significantly lower value of L (Fig. 3). This results demonstrates that, despite the fabric of both the clays is on average well oriented, at different scales, or different magnifications, the fabric may be much less oriented at places, hence is not uniformly oriented at a larger scale. It follows that one-dimensional consolidation, either in the natural site, or in the laboratory, does not bring about a uniformly oriented fabric at the large scale. Rather, it generates a fabric that is on average highly oriented at the medium scale [5, 15, 19], but at the larger scale may be either perfectly oriented (stacks), or not oriented (bookhouse). Further finding of the SEM analyses quoted above is that the natural and the reconstituted clay microstructures, which generate the different compression responses shown in Figure1b, differ much more in bonding than in the type of fabric and fabric orientation degree.

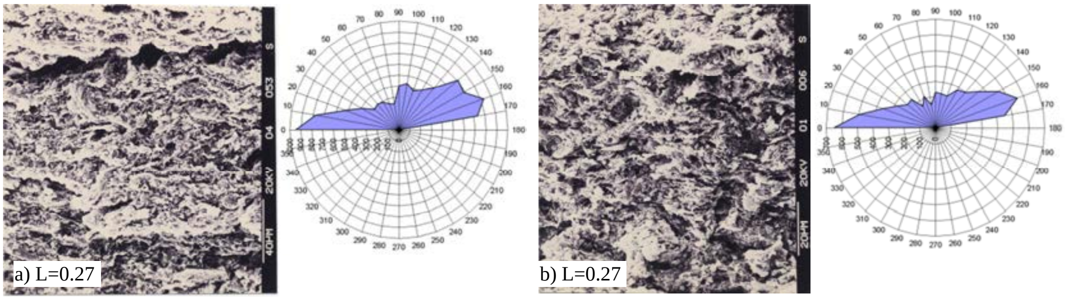
a) L=0.27
b) L=0.27

Fig. 2. SEM on a vertical fracture, direction histogram and index of fabric orientation L of a) reconstituted and b) natural Pappadai clay.

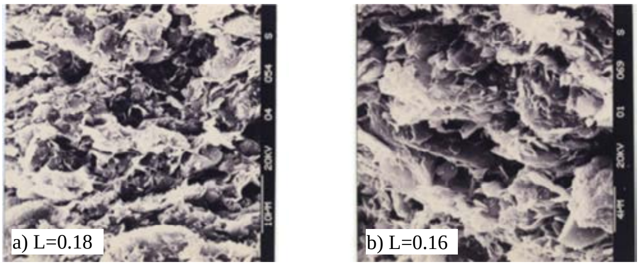
a) L=0.18
b) L=0.16

Fig. 3. Fabric of a) reconstituted and b) undisturbed natural Pappadai clay at high level of magnification and corresponding values of L.

3.3. Microstructural changes generated by compression

In order to investigate the changes in fabric taking place with one dimensional compression, the microstructure of the reconstituted clay at state A* is compared with that at state C* (Fig.1b). The compression to high pressures is found not to give rise to an increase in fabric orientation, the index L remaining almost unchanged (Fig.4a). This suggests that the basic particle aggregation mode and orientation are achieved by the clay already at large water contents and medium pressures [12,15]. For natural Pappadai clay, the undisturbed state (A, Fig.1b) is compared with that immediately post-gross yield (B, Fig.1b), shown in Figure 4b. At gross-yield, the microstructure experiences a major change in fabric, the latter appearing more chaotic, with large pores filled with random fabric aggregates. The orientation of the fabric does not increase much. Conversely, the swelling capacity of the clay increases to the value of the reconstituted clay. Hence, over gross-yield the clay experiences major bonding degradation without significant changes in degree of orientation. At very large pressures (point C, Fig.1b), medium magnification SEM pictures of the natural clay show a completely rearranged fabric, in which stacks of oriented particles appear thicker, but interbed mediumly oriented to honeycomb fabric (Fig.4c, d). Thus, the orientation of fabric of the natural clay does not increase much with compression, the fabric transformation being highly non-uniform. Given the stability of bonding which characterizes the reconstituted clay and the very small changes in fabric orientation, the evolution of the reconstituted clay structure with compression consists mainly in a reduction of porosity, which generates a positive isotropic volumetric hardening, according to critical state soil mechanics. Rather, when the bonding of the natural clay weakens and structure degrades, stress sensitivity decreases, generating a negative component of hardening [2,8,12,18], that affects the state boundary surface.

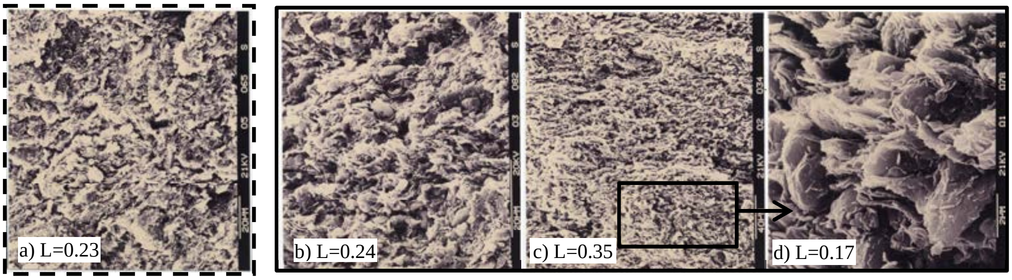

Fig. 4. Fabric of a) reconstituted Pappadai clay (dashed contour) compressed to high pressures and of natural Pappadai (continuous contour) clay compressed b) immediately post-yield and c) to high pressures, with d) detail at higher magnification.

4. Conclusions

The research so far has shown some of the micro-scale features which cause the differences in behavior between natural and reconstituted clay. The diagenetic bonding of the natural clay results in an increase of the gross yield stress and a decrease in the swelling capacity of the clay. With both the clays, one-dimensional consolidation gives rise to a non-uniformly oriented fabric. Despite the degradation of bonding with increasing pressures, the natural structure remains stronger than that of the reconstituted clay, even at high stresses. The statistical analysis of fabric is showing differences between the medium and the high magnifications, hence conveying new knowledge about the micro-processes and features of clays. Further research is on-going, including further mechanical testing and micro-analyses to explore in detail the evolution of fabric, at different states of stress. Further details are likely to be achieved by means of mercury intrusion porosimetry and other explicit image processing techniques.

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