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# Effect of Stress Level on the Microstructural Evolution of Clay under Creep

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Dan ZHAO<sup>1</sup>, Mahdia HATTAB<sup>2</sup>, Pierre-Yves HICHER<sup>3</sup>, Zhen-Yu YIN<sup>4,5</sup>

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Abstract: Creep in clay can significantly affect long-term deformation evolution and 5 6 therefore impact the safety of geotechnical structures. To improve our understanding of the 7 mechanism of creep, we have examined the microstructural evolution of a kaolin clay sample 8 submitted to creep under three-dimensional or axisymmetric loading conditions, focusing on 9 the effect of the stress level. This experimental study identifies the local mechanisms in 10 normally and over consolidated remolded clay samples during creep under triaxial conditions at different stress levels. The results show that the macro and micro behaviors of the kaolin 11 12 clay are predominantly governed by the contractancy/dilatancy mechanism activated along 13 stress paths at constant p'. Within the contractancy domain, the SEM observations showed 14 that the microstructural anisotropy increased with the augmentation of the stress level. The 15 microstructural evolution during creep can be attributed to changing patterns in particle 16 reorientation and pore geometry, resulting in plastic strain hardening/softening as well as in 17 viscous fluid flow. The evolution of the clay microstructure therefore depends on both the 18 stress level and the OCR. The differences in the orientation pattern under creep appeared to be 19 enhanced according to the contractancy/dilatancy mechanism. The dilative specimens 20 exhibited particle orientations which were relatively random. The flattening/expansion of the 21 micropores under creep corresponded to the contraction/dilation mechanism at the specimen 22 scale. An attempt based on the analysis of the SEM photographs was made to evaluate the 23 evolution of anisotropy during the different loading phases.

24 Key words: clay, creep, stress level, triaxial test, microstructure, SEM images, anisotropy

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## 26 Introduction

27 When a soil sample is under a state of constant effective stress, it deforms over time, 28 which corresponds to the phenomenon of creep. Clayey soils usually display significant creep 29 deformation in the form of prolonged settlements of foundations, tilts of geotechnical 30 structures, or slippage of slopes and embankments, for instance (i.e. Mesri & Choi, 1985; 31 Tavenas & Leroueil, 1981; O'Reilly et al., 1991; Rowe & Hinchberger, 1998; Mitchell & 32 Soga, 2005; Karstunen & Yin, 2010; Yin et al., 2015, 2017; Zhu et al. 2014). Generally, the 33 mechanical behavior of clay is determined by the microstructural state of the material and its 34 evolution. (Hattab et al. 2010; Hattab & Favre, 2010; Hammad et al., 2013; Hattab et al., 2015; 35 Xie et al., 2018; Gao et al., 2020; Yu et al. 2019). With regard to the creep behavior, 36 experimental results generally show the development of strains in relation to microstructural 37 changes, this phenomenon depending mainly on the loading history (Mitchell & Soga, 2005; 38 Zhao et al., 2020).

39 On the scale of the specimen, the creep behavior of clay has been widely investigated 40 through triaxial testing (Singh & Mitchell 1968; Bishop & Lovenbury HT, 1969; Mesri & 41 Godlewski 1977; Tavenas et al. 1978; Tian et al., 1994; Zhao et al., 2019). In most of these 42 studies, the effect of stress on the creep behavior of clay was described by time-dependent 43 strain rates, where the axial strain ( $\varepsilon_l$ ) and axial strain rate increased with the stress level. 44 However, the development under creep of the volumetric strain  $(\varepsilon_{\nu})$  appeared to be more 45 complicated. The tests on undisturbed normally consolidated clayey soils under conventional 46 triaxial creep, where the confining pressure  $\sigma_3$  was maintained constant, showed that the 47 volumetric strain  $\varepsilon_{v}$  varied in positive correlation with the stress level as, for example, in the 48 case of marine sediments (Tian et al., 1994). On the other hand, Kong et al. (2011) showed 49 that under higher confining pressure, the volumetric creep strains of undisturbed Zhanjiang 50 clay were quite close to each other at various stress levels. The influence of the stress level on 51 the evolution of the volumetric strain during creep depended on the stress path in the study of 52 Tavenas et al. (1978). By plotting the results in the p'-q plane, they found that an increased p'53 stress path caused an increase of the magnitude of the volumetric strain depending on the 54 stress level; very small volume change was detected under different stress levels along 55 constant p' stress path; an obvious dilatancy occurred continuously and up to failure under 56 high stress level along a decreased p' stress path.

57 To better understand the influence of the loading history on creep behavior, Zhao et al. 58 (2019) performed triaxial creep tests on saturated remolded clay along constant p' stress paths. 59 The results showed that the position of the stress level within the different volumetric domains (defined in the p'-q plane) controls in a significant way the evolution of the 60 61 volumetric creep strain. The different volumetric strain domains were defined by Hattab & 62 Hicher (2004) through purely deviatoric triaxial tests in which a pseudo-elastic zone 63 corresponding to the pseudo-elastic volumetric domain proposed by Biarez & Hicher (1994) 64 was determined. In this domain, the volumetric strain depended only on the variation of p', no 65 volume change was observed during creep (Zhao et al., 2019). Beyond the pseudo-elastic domain, the volumetric strain evolution depended on the components of stress, the deviatoric 66 67 stress q and the mean effective stress p'. Hence, the volumetric strain  $\varepsilon_{\nu}$  increased with the stress level under creep. These results suggest that the effect of stress, *i.e.* the stress level, on 68 69 the macroscopic creep behavior still requires a clearer understanding.

70 At the microstructure scale, the observations usually showed an evolution of the 71 microstructure during creep. Akagi (1994) concluded that creep was the result of the delayed 72 deformation of micropores within the clay particles induced by the viscous flow of the 73 micropore fluid. Sun (2007) stated that the creep of silty clay contains two aspects: particle 74 rearrangement and expulsion of pore fluid from microfabric elements, *i.e.* the closure of the 75 matrix micropores, the generation of microcracks and the expansion of macrocracks as the 76 stress increases. In soft soils, Li et al. (2010) showed a pore variation related to creep, where a 77 gradual change in the micropore properties revealed a micro-mechanism evolution of the clay 78 structure. Xie et al. (2018) investigated the microstructure of a loess-like soil through triaxial 79 creep tests. They stated that the creep was mainly related to the reorientation of particles and 80 pores. They also revealed that the evolution of the meso-pores was the most important factor 81 leading to the creep of these materials. The experimental results recently performed by Zhao 82 et al. (2020) on Kaolin samples showed that the microstructural evolution during creep stages 83 clearly depended on the stress history. Generally, the microstructure variations under creep 84 followed the structural pattern developed under monotonic loading. Their study highlighted a 85 creep dilatancy phenomenon strongly related to the expansion of micro pores and micro 86 cracks within the overconsolidated clay specimens. Nevertheless, the evolution of the clay 87 microstructure under different stress conditions during creep is still under discussion.

This study focuses on two specific aspects of creep development after purely deviatoric stress paths: *i*. the effect of the stress level along a given constant p' stress path; *ii*. the effect of the stress level at a constant stress ratio. Significant insight comes from analyzing the microstructure evolution along the different loading stages. The experimental approach 92 consists of performing a series of triaxial creep tests and identifying the microstructure, 93 particle orientations, and pore properties along the creep stage. The latter analyses were 94 quantified using the scanning electron microscopy (SEM) technique on different samples after 95 triaxial loading. The local mechanisms explaining the development of the creep process in 96 relation to the stress state were revealed by linking the microstructural characteristics to the 97 mechanical behavior.

98

# 99 Material and experimental techniques

#### 100 Material and triaxial test method

101 The soil selected for this research is an industrial Kaolin K13 clay whose liquid limit is 102 42%, plastic limit is 21% and specific gravity is 2.63. The compression index is 0.288 and the 103 swelling index is 0.085. According to the measured grain size distribution, the mean 104 dimensions of the clay "grains" fall into the range of 2.5  $\mu$ m to 20  $\mu$ m, which may concern 105 both unit particles as well as aggregates of larger sizes. For the mechanical tests, cylindrical 106 specimens, 75 mm in height and 50 mm in diameter, were cut from a one-dimensional pre-107 consolidated core.

108 The triaxial tests were carried out on saturated remolded kaolin specimens by using the 109 GDS triaxial testing system. The tests included two categories: the triaxial shear group and the 110 triaxial creep group. These two groups of tests were conducted following the same saturation, 111 consolidation and shearing steps. Along with the triaxial shearing procedure, constant p'112 triaxial stress paths were applied up to a given stress level. The second group consisted in a 113 series of creep tests performed at the same stress level. Both p' and q were kept constant for at 114 least 7 days to measure the creep deformation. After the triaxial loading, all specimens were 115 unloaded by steps. After having been retrieved from the cell, the specimens were enveloped in 116 film paper, tin foil and paraffin, and then kept at constant temperature before being prepared 117 for SEM analysis. Let us note that one can find the procedure of the sample preparation and the mechanical test principle described in more details in Zhao et al. (2019). The specification 118 119 of the triaxial tests is summarized in Table 1. The specimen denomination corresponds to the 120 loading condition. For example, for P<sub>010</sub>-OCR4-q200, P<sub>010</sub> represents the consolidation stress 121  $p'_{0i} = 1000$  kPa; OCR4 represents an overconsolidation ratio equal to 4; the NC symbol 122 represents normally consolidated specimens; q200 indicates that a deviatoric stress of 200 kPa 123 was applied during constant p' triaxial shearing. The subscript "shear/creep" represents the 124 two categories of tests.

125 The loading state at the end of the shear/creep for these two groups of triaxial tests is 126 shown in the (p'-q) plane in Fig. 1. In this plane, the contractancy, no-volume change (pseudo-127 elastic), and dilatancy domains identified by Hattab & Hicher (2004) were superimposed 128 (Zhao et al., 2019). In this study, we performed tests under stress conditions marked by 129 numbered lines in Fig. 1. Line ① represents two tests having the same OCR under different 130 stress levels (named SL) and belonging to the contractancy domain; line 2 represents two 131 tests under different SLs with the same OCR, one within the contractancy domain and the 132 other within the no-volume change domain. The two series of tests aimed to analyze the role 133 of the stress level; line ③ corresponds to the three tests located along a constant stress ratio 134 (named  $\eta$ , where  $\eta = 0.67$ ) line, but with different OCRs, thus different history of loading of 135 the samples, their loading paths evolving within the contractancy or dilatancy domain.

### 136 Microscopic identification techniques

### 137 Specimen preparation

138 The preparation of an undisturbed small sample for microstructure observation plays a key 139 role in this microscopic study. After triaxial testing, the core zone of each cylindrical 140 specimen was cut into a soil board to a dimension of 30 mm  $\times$  30 mm  $\times$  10 mm. Thereafter, a 141 sub-specimen of 10 mm in height was extracted from the soil board. The surface layer of the 142 sub-specimen was notched slightly in the middle section in order to obtain an undisturbed 143 observation plane. Note that the length direction of the sub-specimen was referenced as 144 perpendicular to the axial stress ( $\sigma'_1$ ) applied to the cylindrical specimen in the triaxial cell. 145 Specific preparation permitting to preserve the clay fabric of the sub-specimens were carried 146 out before introducing them inside the SEM cell (Delage & Pellerin, 1984; Hattab et al. 2010; 147 Hammad et al. 2013; Gao et al. 2020; Zhao et al. 2020). With the following stages of sub-148 specimen preparation, a dry and clean section for identifying the microstructure of the kaolin 149 clay by SEM could be obtained: *i*. dehydration through the freeze-drying method; *ii*. 150 fracturing stage to obtain a fresh section for the observation; *iii.* metallization of the section 151 with gold. It should be noted that in previous studies, the SEM images with magnifications of 152  $1000 \times$  to 5000 × were suitable for the identification of the clay microstructure (Hattab et al., 153 2010; Zhang & Cui, 2017; Gao et al., 2020 and Zhao et al., 2020). Thus, 3000× magnified 154 images were selected for this research. For a representative result to be obtained, a large 155 number of images is required for a statistical interpretation. Around 20 typical areas were

156 randomly selected on each observed surface for each specimen.

#### 157 Image processing methods

158 Kaolinite particles are shaped as rigid platelets that can form parallel-stacked mineral layers 159 (Fig. 2a). From the view of the vertical plane, the kaolin particles that contain the orientation information exist as ellipsoid flakes. Thus, the particles can be simulated one by one through 160 161 short lines that have the same length and the same orientation as the major axis length and 162 orientation of the ellipsoid flakes (Fig. 2b). Let us note that the axisymmetric loading 163 condition of the specimens implies that the particles seen by face, which are parallel to the 164 image, correspond to the particles of the surface seen by their edge. Thus, they are not 165 simulated. The orientations of the represented lines with respect to the X-axis can be 166 measured by the angle  $\theta$  (0°~180°), where  $\theta$  equal to 0°/180° means that the particle is perpendicular to  $\sigma'_{1}$ , see the illustrations in Fig. 2c. The imaging plane was divided into 12 167 168 quadrants, each of which equals to 15°. The number and the percentage of particles oriented towards each quadrant were calculated, represented by the orientation curve in Fig. 2c. The D 169 170 line in Fig. 2c, with a mean percentage of 8.3%, represents a perfectly isotropic 171 microstructure (Hattab & Fleureau, 2011).

172 In Fig.2a, the raw micrograph obtained by SEM is represented by the greyscale image. 173 Generally, the SEM image presents the pore spaces in a darker greyscale compared to the 174 solid particles. Thus, the pore space and the solid particle can be discriminated. In this study, 175 the pore properties (orientation and dimension) were partly identified by the Image J software. 176 The original image was transformed into the binary image (Fig. 2d) by setting an optimal 177 threshold value. Then, the connected pores were separated through the distance transform and 178 watershed segmentation algorithm. Afterwards, using an internal Image J feature, the 179 segmented pores were fitted with ellipses, keeping as much as possible identical areas, aspect 180 ratios, orientations and centroids (Fig. 2e). The detailed identification method refers to the 181 works of Hicher et al. (2000), Hattab & Fleureau (2010), Gao et al. (2020) and Zhao et al. 182 (2020).

After the identification of particles and pores, different local parameters were deduced: the orientation index  $I_{or}$  (Eq. 1) which expresses the anisotropy of the structure (Hicher et al., 2000) and the pore roundness  $R_s$  (Eq.2) which characterizes the shape of a pore.

186 
$$I_{\rm or} = \frac{k+l+m}{r+s+t} \tag{1}$$

187 
$$R_{\rm s} = \frac{B}{A}$$
 (2)

Where  $I_{or}$  varies from 0 (for a totally anisotropic arrangement) to 1 (for an ideal isotropic structure); *s* is the maximum percentage (the peak value in Fig. 2c); *r* and *t* are the percentages of the two zones beside the maximum one; *k*, *l*, *m* are the percentages of the zones perpendicular to the zones *r*, *s* and *t*, respectively.  $R_s$  varies from 0 (for a very elongated pore) to 1 (for an equiaxed pore); *A* and *B* are the major and minor axis length of the fitted ellipse, respectively.

194

# 195 Strain mechanisms under creep and microstructure features

196 The strain mechanisms of kaolin specimens under a pure deviatoric stress path have been 197 widely discussed by Hattab & Hicher (2004) and then by Zhao et al. (2019, 2020). The latter 198 framework is taken in this study to discuss the influence of the stress level on creep after 199 purely deviatoric paths, as well as the influence of the stress history by keeping constant the 200 stress ratio. Let us consider the representative groups of NC tests and the tests with OCR=1.5 201 represented by line ① and line ② (Fig. 1), respectively. For line ②, the two stress levels are 202 located in two different volumetric domains: the pseudo-elastic domain for the lower stress 203 level (q=200 kPa), and the contraction domain for the higher stress level (q=445 kPa).

In what follows, the analyses are focused on both the macroscopic behavior and the obtained microstructure features.

#### 206 Tests on normally consolidated samples

207 For normally consolidated (NC) samples under loading paths within the contractancy 208 domain, a contractant behavior, as expected, was detected from the beginning of the 209 monotonic loading (Fig. 3) and the compression was more pronounced under a higher stress 210 level. The experimental results show a good consistency within the same loading interval, 211 which permits to directly identify the mechanisms of evolution related to the stress level. 212 During creep, a contractive behavior was also obtained (Fig. 4a) and the contractive creep 213 deformation was larger under a higher stress level. The evolution of the volumetric creep 214 strain ( $\varepsilon_{v-creep}$ ) under a purely deviatoric stress path for NC samples is consistent with the 215 results under constant  $\sigma'_3$  by Tian et al (1994), and those under an increasing p' stress path by 216 Tavenas et al. (1978) and by Sekiguchi (1973). Fig. 4b shows that a higher stress level produced a relatively larger volumetric strain rate, but the difference tended to becomenegligible after about 1000 minutes in the creep phase.

219 Fig. 5 presents representative SEM photos of the test P<sub>010</sub>-NC-q670 within the 220 contractancy domain after shear and after creep. One can see that the particles were mostly 221 arranged face-face along oriented planes and reoriented by groups towards a given orientation 222 after creep (Fig. 5b) in a more pronounced way than after the monotonic shear loading (Fig. 223 5a). The preferential microfabric of clay along purely deviatoric stress path is quite different 224 from the random orientation pattern identified after isotropic consolidation, see for instance 225 Hicher et al. (2000), Hattab & Fleureau (2011) and Gao et al. (2020). This highly oriented 226 microstructure combined with its densification appears to be directly related to the contractive 227 behavior at the specimen scale.

#### 228 Slightly overconsolidated case - OCR=1.5 samples

#### 229 Mechanical behavior analysis

The stress-strain relations for OCR=1.5 samples under monotonic loading are shown in Fig. 6. The P<sub>010</sub>-OCR1.5-q445<sub>shear</sub> test showed a clear and very marked contractancy starting at q = 227 kPa, which was very close to the limit of the pseudo-elastic domain. The path evolved afterward within the contractancy domain, reaching a volumetric strain of  $\varepsilon_v$ = 0.58% at the stress level of q = 445 kPa. For the four shear tests, no significant volumetric strains were observed, given the accuracy of the experimental system, when the paths evolved within the pseudo-elastic domain.

237 The evolutions of the volumetric creep strains as a function of time are presented in Fig. 238 7a. For specimens located within the pseudo-elastic domain ( $P_{010}$ -OCR1.5-q200<sub>creep</sub>), the 239 result showed that a volumetric strain evolution alternated between very small contractancy 240 and very small dilatancy. These results seem to be directly related to the constant stress 241 control condition, thus it is reasonable to assume that no volumetric strain under creep 242 developed within this pseudo-elastic domain (Zhao et al., 2019). For soil creeping within the 243 contractancy domain, P<sub>010</sub>-OCR1.5-q445<sub>creep</sub>, the contractancy was more pronounced and 244 developed continuously. A linear tendency of the volumetric creep strain rate was observed after about 1000 minutes (Fig. 7b), corresponding to the well-known primary creep. Fig. 7b 245 shows that, in the early period of creep, the relationship between  $log \varepsilon_{v-creep}$  and  $log t_{creep}$  is 246 curved for the three specimens within the contractancy domain. Afterward, the volumetric 247 248 strain rates varied inside a quite narrow band after  $t_{creep}=1000$  minutes. Let us note that Tian et al. (1994) obtained similar tendencies on normally consolidated marine sediments from theGulf of Mexico and Zhu (2007) on Hong Kong marine sediments.

#### 251 *Microstructure features*

Fig. 8 shows the representative SEM photos for the samples sheared under different stress levels, whereas Fig. 9 shows the representative images after creep under different stress levels.

254 In Fig. 8 the vast majority of isolate particles is plate-shaped and form aggregates with a 255 maximum diameter of about 10  $\mu m$ . The mean size of an individual particle is about 1.5  $\mu m$  in 256 length. Generally, the particles are face-face associated, as shown by thin layers in the side 257 view of the SEM photos. In Fig. 8a, one can see that the groups of particles are partly 258 arranged edge-face and partly face-face. These results are similar to what was obtained for 259 specimens with OCR=2.5 located within the pseudo-elastic domain (Zhao et al., 2020), which 260 suggests that similar microstructures are related to this particular domain. With the increase of 261 the stress level within the contractancy domain, a clear contractancy developed at the sample 262 scale resulted in an obvious preferential oriented pattern at the microscopic scale (Fig. 8b). 263 The particles were mostly face-face along certain preferential oriented planes, showing an 264 anisotropy which was also observed by Hattab & Fleureau, (2011). Globally the system here 265 shows a trend towards a denser structure. Thus, rearrangements in the clay microstructure 266 developed with the increase of the stress level along purely deviatoric monotonic loading.

In the case of the  $P_{010}$ -OCR1.5-q200<sub>creep</sub> specimen within the pseudo-elastic domain, the microfabric tended to evolve isotropically rather than anisotropically after a certain creeping time (Fig. 9a), similarly to the state of the structure for the specimen after shear (Fig. 8a). As for the  $P_{010}$ -OCR1.5-q445<sub>creep</sub> specimen under a higher stress level within the contractancy domain, the microstructure rearranged towards an apparent anisotropy. Obviously, these results highlight two different features depending on the stress level, as well as on the domain where the creep occurred.

274

# 275 Creep phenomenon - Microstructural study

Following the experimental procedure described in *Section 2.2.2*, the evolution of the particle orientation and the pore properties related to different loading histories will be examined in this section.

#### 279 Influence of stress level under the same OCR

#### 280 Clay particle orientation mechanism for NC samples

Fig. 10 presents the global particle orientation curves of 4 normally consolidated samples 281 282 under different stress levels located within the contractancy domain, following the condition 283 of line (1) (Fig. 1). The global orientation of the clay particles is illustrated in the form of 284 orientation curves. The oriented curves of specimens at q=200 kPa and 670 kPa after shear 285 were drawn from 10 photos representing 4347 particles and 14 photos representing 7156 286 particles, respectively (Fig. 10a). The curves for the two specimens after creep were obtained 287 from 14 photos representing 7227 particles and 19 photos representing 7170 particles, 288 respectively, as shown in Fig. 10d.

289 Fig. 10a shows that the orientation curves for the two sheared samples within the 290 contractancy domain depart from the structural isotropic D line, as identified on the same clay 291 after an isotropic loading by Gao et al. (2020). The peak values for the percentage of particle 292 orientation of the two specimens fall into the range of 150° to 165°, which means that the 293 majority of individualized particles and particle groups are oriented in this direction (as 294 illustrated on the representative image in Fig. 5a). This result observed at the local level 295 demonstrates the existence of an anisotropic microfabric, which corresponds to the 296 contractancy at the macroscopic level of the specimen. Furthermore, with larger compression 297 of the specimen under a higher stress level, more obvious structural anisotropy can be seen, 298 the orientation curve being further apart from the D line (Fig. 10a).

299 In Fig. 10b and Fig. 10c, the microstructure analyses show that the orientation curves 300 exhibit more marked peaks after creep than after shear. For the two samples after creep, the 301 orientation angles are quite similar to those obtained after shear tests, but with higher 302 magnitudes (Fig. 10d). These results suggest that particle rearrangement continues during 303 creep, with more particles joining the preferential direction, as shown in Fig. 5b. The peak 304 value **P** in Fig. 10 indicates that the principal orientation is located between  $150^{\circ}$  to  $165^{\circ}$ 305 under two stress levels. This can be attributed to the induced anisotropy developed during 306 creep. It is interesting to note in Fig. 10d that the particle orientation for samples after creep 307 seems to be independent of the stress level. This phenomenon may highlight a dominating 308 contractancy mechanism related to creep within the volumetric contractancy domain.

309 At the microscopic scale, the NC specimens show the development of the anisotropy 310 combined with densification at the macro scale, the majority of the particles being oriented in 311 a preferential direction. These results are in agreement with those previously obtained under monotonic loading for instance by Hicher et al. (2000), Hattab et al. (2010) and more recently Gao et al. (2020). During creep, the anisotropy continues to evolve, resulting in the increase of the number of particles oriented in a preferential direction (see the example of test  $P_{010}$ -NCq670 in Fig. 5).

#### 316 Clay particle orientation mechanism for OCR=1.5 samples

317 A qualitative analysis of the photos taken at different observation points for the lightly 318 overconsolidated specimens is shown in Fig. 11. The oriented curves at q=200kPa and 319 445kPa after shear were drawn from 11 photos (5639 particles) and 14 photos (7156 particles), 320 respectively (Fig. 11a). By comparing the two curves, it appears that the curve at q = 200 kPa 321 has moved closer to the perfect isotropic D line, whereas at q = 445 kPa, the curve has 322 departed away from the D line with an obvious peak value in the 150° to 165° direction (point 323 **P** in Fig. 11a), which means that the majority of the individualized particles are oriented in 324 this direction (see the representative image in Fig. 8b). The global orientation curves of the 325 clay particles clearly show the influence of the stress level during the monotonic loading. For 326 a clay specimen under a lower stress level located within the pseudo-elastic domain, the 327 preferential reorientation of the clay particles is not dominant; it seems that the initial 328 isotropic microstructure developed during isotropic consolidation has not been completely 329 erased. Under higher stress level within the contractancy domain, an anisotropic orientation 330 tends to develop along the purely deviatoric loading phase, resulting in a large number of clay 331 particles rotating towards a preferential direction.

332 Fig. 11b and 11c present the particle orientation before and after creep under different stress 333 levels. It can be seen in Fig.11b that, for the two samples loaded within the pseudo-elastic 334 domain, a slight change in particle orientation was identified after creep. The limited particle 335 orientation evolution during creep would not introduce an obvious macroscopic deformation. 336 The results for the test P<sub>010</sub>-OCR1.5-q445 are abnormal compared to the continuous 337 contractancy at the sample scale (Fig. 7a). Less structural anisotropy was identified after creep 338 than after shear. This abnormal experimental result may be due to the observation spots on 339 SEM images of the specimen P<sub>010</sub>-OCR1.5-q445<sub>shear</sub>; this aspect will require further 340 experimental verification. It should be noted that the results of P<sub>010</sub>-OCR1.5-q445<sub>shear</sub> give at 341 least a reasonable structural anisotropy. Thus, the results of this test were maintained without 342 any strong conclusion being drawn from them.

Fig.11d compares the global particle orientation of the lightly overconsolidated specimens
 after creep under two different stress levels. The shapes of the orientation curves appear quite

345 consistent with those after shear (Fig.11a). This may demonstrate that the particle 346 rearrangement during creep follows the same structure pattern as the one formed in the 347 monotonic loading stage. For the sample at q = 200kPa creeping within the pseudo-elastic 348 domain, very limited structure rearrangement took place during creep. The microstructure 349 evolution is in agreement with the findings obtained at the macroscopic scale, showing that no 350 volumetric strain took place along the p' constant stress path. For the sample at q = 445 kPa 351 creeping within the contractancy domain, the dominated direction (around 160°) obtained for 352 the sample after shearing (Fig.11a) was maintained.

### 353 Pore property evolution for NC samples

The particle orientation modes have to be associated with the pore space and its geometric distribution including orientation, roundness and elongation. The coupling of both systems can define the state of the material at the representative elementary volume scale.

The particles rearranged during loading, which resulted in a certain preferred orientation; consequently, the corresponding inter-particle pore geometry can provide complementary information on the structural rearrangement. Generally, the global pore orientation is consistent with the particle orientation identified in *Section 4.1.1*, as shown in Gao et al. (2020) and Zhao et al. (2020). For these tests following the same p' constant stress path, the distribution pattern of pores depends on the magnitude of the stress applied to the specimen.

363 For the two normally consolidated samples sheared within the contractancy domain, the 364 global pore orientations under different stress conditions (Fig. 12a) are quite consistent, as 365 expected, with the particle orientations (Fig. 10a). After the monotonic loading, the structural 366 anisotropy increased under a higher stress level (Fig. 12a), resulting in a curve clearly further 367 from the isotropic  $D_{pore}$  line. The  $D_{pore}$  line in terms of pore orientation has a similar meaning 368 as the depolarization line (D line) of particle orientation. However, a slightly weaker 369 anisotropy of the micro pores for the specimen after creep under higher stress level could be 370 observed (Fig. 12b), which may indicate that a specific mechanism controlled the pore 371 evolution under creep.

As illustrated in Fig.2, the pores identified in SEM images can be fitted by ellipses. The shape characteristic of the pores can thus be evaluated through the roundness of the fitted ellipse by Eq.2. Figs. 13-14 compare the global particle shapes of four normally consolidated specimens under different stress levels. All the roundness curves show unimodal distributions 376 with a marked peak of a dominant roundness relatively distant from Rs = 1 (Figs. 13a & 14a), 377 which indicates that most pore shapes are clearly elongated.

378 After the monotonic loading, the mean lengths of the pores increased with the stress level (Fig. 13b), with  $L_{mean}$  = 0.566 µm under q = 200 kPa and  $L_{mean}$  = 0.713 µm under q = 445 kPa; 379 380 whereas the mean diameters showed an opposite trend, with  $D_{mean} = 0.284 \ \mu m$  under q = 200381 kPa and  $D_{mean}$  = 0.142 µm under q = 445 kPa (Fig. 13c). These results indicate that the pores were elongated as well as flattened with the increase of the specimen contraction developed 382 383 under a higher stress level. During the creep phase, taking the example of test P<sub>010</sub>-NC-q200, 384 the pores tend to be flatter after the creep stage, illustrated by a smaller mean pore diameter in 385 the specimen after creep (Fig. 14c) than in the specimen after shearing (Fig. 13c), whereas the 386 pore length before and after creep remained relatively unchanged (Fig. 13b &14b). In the case 387 of the specimen under a higher stress level, the mean pore length was shortened after creep. 388 Furthermore, the mean pore parameters for the two creep specimens appear quite similar (Fig. 389 14) regardless of the difference in the stress level. The same consistency can be seen in the 390 particle orientation in Fig. 10d, which highlights a dominating contractancy mechanism 391 related to creep in this volumetric domain.

#### 392 Pore property evolution for OCR=1.5 samples

In the case of lightly overconsolidated samples, the structural anisotropy increased under a higher stress level (Fig. 15a), resulting in a curve clearly further from the isotropic  $D_{pore}$  line. After a certain duration in the creep phase, the differences in the orientation of pores under different stress levels decreased (Fig. 15b). The structural isotropy evolved for the specimen under q = 445kPa, whereas a structural anisotropy developed for the sample under q = 200kPa, resulting in a final orientation close to the horizontal.

399 The statistical results presented in Fig. 16 bring more insight. The pore shape variations of 400 four lightly overconsolidated specimens after shear and after creep are shown. Compared to 401 the curves drawn from the four NC samples, the differences in the pore shapes with OCR = 402 1.5 are more significant. In Fig. 16a, it can be seen that the mean roundness for the 2 403 specimens under q = 445kPa (Rs = 0.45) is smaller than for the 2 specimens under q = 200kPa 404 (Rs = 0.55). This means that the pores tend to be generally smaller under higher stress levels, 405 as shown by a clearly lower percentage of the mean length and mean diameter in Fig. 16b and 406 16c. The variation of the pore shape with SL is reflected especially in the change of the mean 407 diameter (Fig. 16c). It can be seen that the difference in pore diameter between the two 408 specimens sheared under two different stress levels is mainly in the range of D>0.28 µm, which indicates that the largest pores are more likely to contract with the increase of the stresslevel, resulting in a denser structure.

For the two P<sub>010</sub>-OCR1.5-q200 specimens within the pseudo-elastic domain, no volume change occurred at the macroscopic level under the creep phase (see Fig. 7a). The mean pore shape is the same for the sheared and the creep specimens for which  $Rs_{mean}$  is 0.55,  $L_{mean}$  and  $D_{mean}$  is 0.713 µm and 0.357 µm, respectively. The phenomenon appears similar to what was observed for an OCR=2.5 specimen also located within the pseudo-elastic domain (Zhao et al., 2020).

For the two P<sub>010</sub>-OCR1.5-q445 specimens within the contractancy domain, the change of the pore shape during the creep phase concerns mainly the pore diameter, with a higher percentage of pores for D>0.284 $\mu$ m and a smaller percentage of pores for D<0.284 $\mu$ m in the creep specimen than in the sheared specimen (Fig. 16c). This result may indicate that the contraction under creep occurs mainly by the flattening of the smaller pores.

#### 422 Microstructures under same stress ratio but different loading histories

For specimens under a stress level located on the line ③ (Fig. 1), corresponding to a stress ratio  $\eta = 0.67$ , the results show a contractancy for both the NC and the OCR=1.5 specimens, whereas for the highly overconsolidated clay a dilatancy was detected at the first stage of the monotonic loading. During the creep phase, contractancy or dilatancy developed continuously as can be observed in Fig. 17a, the volumetric creep strain rate (the absolute value of  $\varepsilon_{v-creep}$ was introduced for the case of OCR=4) decreasing with the increase of OCR in the early stage of the creep test, and then tending to be similar at the later stage of the creep test (Fig. 17b).

# 430 Clay particle orientation mechanism

The particle orientation curves for the three specimens are shown after shearing in Fig. 18a and after creep in Fig. 18b. The shapes of the orientation curves for the two stress levels located within the contractancy domain are quite consistent, showing both a clear structural anisotropy development. For the highly overconsolidated specimen ( $P_{010}$ -OCR4-q200)<sub>shear</sub>, located within the dilatancy domain, the orientation curve moves towards the D line, meaning that the microstructure tends towards a structural isotropy. The isotopic microstructure appears here related to the dilatant behavior at the macroscopic scale.

438 For the three specimens after creep (Fig. 18b), the differences are increased, due to the 439 particle rearrangement under the creep phase with different stress paths. The percentage of particles in the privileged direction (150° to 165°) decreased with OCR, meaning that the
induced anisotropy or isotropy tended to increase.

#### 442 Pore property evolution – Orientation, roundness and elongation

As expected, the evolution of the pore orientation appears to be consistent with the particle orientation. A significant influence of the OCR on the pore orientation, linked to the dilatancy mechanism, could be observed. As shown in Fig. 19a, the dilative specimen  $P_{010}$ -OCR4-q200<sub>shear</sub> had smaller maximum percentages, which indicates that more random micropore orientations developed within the specimen during loading. The contractancy or the dilatancy continued to develop during the creep phase, resulting in the orientation curves to approach the  $D_{pore}$  line with the increase of OCR as can be observed in Fig. 19b.

450 The statistical results in Fig. 20 show the distribution of the pore shapes for the three 451 specimens under different OCRs after the creep phase, the stress levels being located along 452 the constant stress ratio line  $\eta = 0.67$ . Altogether, as shown previously in Fig. 16, the creep 453 stage led to an extension of the tendencies activated by the shear loading stage. For the 2 454 specimens creeping within the contractancy domain (NC and OCR=1.5 samples), the pore 455 shape tended to show very similar roundness distribution curves (Fig. 20a). However, for the 456 highly overconsolidated specimen with OCR = 4 located within the dilatancy domain, the 457 curve shifted to the right, highlighting globally rounder pores. The influence of the stress path 458 on the evolution of the pore shape under creep pertains mainly to the evolution of the pore 459 diameter: a clear increase in the mean pore diameter when OCR increased from 1 to 4 could 460 be observed in Fig. 20c. The opening/expansion of the micro pores associated with a 461 depolarization orientation contributes here to the dilatancy mechanism at the sample scale.

## 462 **Discussions**

Based on the current statistical results of particles and pores properties, the evolution of 463 464 the clay microstructure is clearly dependent on the stress history, as well as on the stress level in the triaxial loading stage. In the creep stage, the development of contractancy/dilatancy at 465 466 the sample scale is accompanied by a continuous rearrangement of the soil structure. In what 467 follows, the microstructural evolution related to the creep mechanism will be discussed in 468 relation to the stress level influence. The illustrations of the conceptual models within 469 different volumetric strain domains under different stress conditions are shown in Fig.22, 470 where the black dotted lines represent the orientation of groups of particles and the blue 471 circles represent the state of the pores.

472 Within the contractancy domain for normally consolidated samples, the depolarization (or 473 the isotropic microstructure) first induced by the isotropic triaxial loading was progressively 474 erased along the purely deviatoric stress path (see the sketches within the contractancy 475 domain after shear in Fig.22). The influence of the deviatoric stress seems predominant in the 476 particle rotation mechanism towards privileged orientations. When the stress level increase 477 leads to larger strains, the rotation of the particles becomes more apparent and their 478 orientation progressively tends towards privileged orientation lines of 150° to 165°. The 479 rearrangement of the soil structure under stress accompanied by the free water flowing out of 480 the soil element induces the decrease of  $D_{mean}$  along with the increase of  $L_{mean}$  (Fig. 21b and 481 Fig.22). Consequently, the contacts between particles gradually increase from a random edge-482 face contact after isotropic consolidation to a face-face contact along monotonic loading. The 483 specimen experiences a denser structure with the increase of SL in this domain governed by 484 the contractancy mechanism. In the creep phase, plastic strains developed at the macro scale. 485 At the microscopic scale, the deformation of the pores either in length or in diameter (Fig. 21b 486 and Fig.22) and, as a consequence, the drainage of the pore fluid, led to an increase of the 487 induced anisotropy, *i.e.*, the decrease of the orientation index *I*<sub>or</sub> (Fig. 21a).

488 For the lightly overconsolidated samples loaded from the pseudo-elastic domain up to the 489 contractancy domain, two different orientation modes could be characterized (see the sketches 490 within the pseudo-elastic domain after shear in Fig.22). At the end of the monotonic loading, 491 the particles of the specimen remaining within the pseudo-elastic domain are mainly 492 associated by groups oriented along oblique planes. The development of this structural mode 493 is assumed to be related to the very light contractancy phenomenon in monotonic loading 494 under low stress levels, where the isotropy is still dominant. During creep, a null volumetric 495 change is assumed at the macroscopic scale. At the microscopic scale, the particle orientation 496 and especially the porosity do not evolve significantly in the creep phase (Fig. 22b). The quite 497 limited macroscopic deformation alternating between contractancy and dilatancy was found to 498 be provoked by the slight fluctuation in the GDS system trying to maintain a constant stress 499 condition (Zhao et al., 2019). The microfabric under the fluctuating stress/strain corresponds 500 to a progressive adjustment of the clay structure to reach equilibrium under the external 501 effective stresses.

However, when the stress level reaches the contractancy domain, the contractancy mechanism is activated; the clay particles are mainly associated face-to-face and oriented in an oblique direction mode (Fig. 22). Although a continuous contractancy of the lightly 505 overconsolidated sample can be observed, the microstructure evolution under creep is, in this 506 case, less pronounced than in the NC samples. A decrease of anisotropy for the specimen after 507 creep was obtained (Fig. 23a); however, the porosity evolution remained quite limited (Fig. 508 23b). It may be assumed that a weakened or delayed mechanism exists due to the loading 509 history within the pseudo-elastic domain, which requires deeper analyses.

510 The experimental tests conducted in this study aimed at contributing to a better 511 understanding of the micro-macro mechanisms in clay related to creep. According to the 512 analysis of the microstructure through SEM images, the creep mechanism can be seen as the 513 displacement of groups of clay particles, called aggregates. The relative sliding occurs mainly 514 between adjacent groups of particles. A promising approach to the micro-macro constitutive 515 modelling is to treat clay as an assembly of aggregates interacting by means of contact 516 (normal and shear) forces and to attempt to derive stress-strain relationships on this basis. The 517 contact forces, together with the contact orientation of the particles, constitute the fabric of a 518 clay sample.

The orientation distribution of the particle deposition angles was obtained through SEM images. For a contact between two particles, the contact orientation is defined as the normalized vector connecting the centroids of two particles, as shown in Fig. 24 in the local coordinates, where  $\theta$  and  $\beta$  are the angles of inter-particle contact, *n*, *s*, and *t* represent the components of the local forces in the three directions of the local coordinate system. For a particulate assembly, the contact orientation distribution can be characterized by an angular distribution  $E(\theta)$ , which is given by:

526

$$E(\theta) = \frac{1}{4\pi} [1 + a\cos 2(\theta - \theta_a)]$$
(3)

where a defines the magnitude of anisotropy and  $\theta_a$  defines the direction of anisotropy 527 528 (Rothenburg & Bathurst, 1989; Wang & Yin, 2020). The parameter a can be fitted from the contact orientation distribution using Eq.(3). When a = 0 the distribution is perfectly isotropic. 529 530 The values of the fabric parameter *a* obtained after shear and creep are summarized in Fig. 25. 531 For the isotropically loaded sample, a value of a close to zero was obtained. For samples 532 located within the contractancy/dilatancy volumetric domain, the magnitude of anisotropy a 533 increased/decreased along the creep phase, resulting in a higher/lower *a* value for the samples 534 under creep than that for the samples under shear. Meanwhile, the changes of  $\theta_a$  in these two 535 zones indicate that the direction of anisotropy also evolved during creep. It should be noted 536 that for samples located within the pseudo-elastic volumetric domain (P<sub>010</sub>-OCR1.5-q200), the 537 direction of anisotropy  $\theta_a$  remained almost unchanged after shear and after creep, while the 538 magnitude of anisotropy *a* increased to some extent. The calculated parameters in Eq.(3) 539 match well the experimental observations.

540 Based on SEM photos from different loading stages corresponding to different stress 541 states and plastic strains, the fabric evolution can be characterized as a function of stresses and 542 plastic strains. The equation of a and  $\theta_a$  can then be applied to micromechanical/multiscale 543 models, such as the recently developed micromechanics-based elastoplastic model for 544 granular materials, clays and soil mixtures by authors (Chang & Hicher 2005, Yin et al. 2009; 545 Yin et al. 2014; Zhao et al. 2018), since these kind of models use the orientation distribution 546 function of contacts to consider the soil anisotropy. Our ultimate objective is to formulate the 547 fabric tensor as a function of the macroscopic strain/stress tensor relating to creep. The 548 development of such model is in progress and will be the topic of a future paper.

549

## 550 Conclusion

551 This study attempted to characterize the microstructure of remolded kaolin subjected to 552 various triaxial stress paths followed by a creep stage. Ten triaxial tests were performed on 553 both normally and overconsolidated samples under different stress levels and stress ratios. 554 Afterward, the SEM technique was used to identify the microstructural features of the clay 555 before and after creep. From these analyses, the following points can be observed:

- The evolution under creep depends on the deformation at the end of the monotonic
  loading. The microstructure pattern of particles and pores identified through SEM images
  showed results that are consistent with the tendencies at the sample scale.
- The influence of the stress level on the creep deformation in normally consolidated samples followed the same pattern as what was obtained along monotonic loading paths.
  For normally consolidated specimens within the contractancy domain, the particles and pores showed preferential orientation, indicating the development of a structural anisotropy and this tendency continued during creep. The increase of the stress level led to a decrease of the pore diameter after creep, resulting, from plastic strain hardening as well as viscous flow, in a more stable structure.
- The influence of the stress level on the creep deformation of lightly overconsolidated
   clay depended on the position of the stress state within a given volumetric domain.
   Structural anisotropy and decrease of pore diameter were also obtained for a lightly

569 overconsolidated specimen within the contractancy domain under a high stress level. 570 However, for a lightly overconsolidated specimen under a lower stress level within the 571 pseudo-elastic domain, the clay structure was in an intermediate state, the microstructural 572 evolution during creep in this domain being quite limited.

The changes in loading history and variations in both stress level and OCR provoked different behaviors. The dilative specimens exhibited relatively random orientation. The differences in the orientation pattern appeared enhanced in agreement with the contractancy/dilatancy mechanism during creep. The flattening/expansion of micropores under creep corresponded to the contraction/dilation mechanism at the specimen scale within the different volumetric domains.

# 579 Data availability statement

All data that support the findings of this study are available from the corresponding authorupon reasonable request.

582

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# **Table**

# 

Table 1 Triaxial tests on Kaolin K13 clay

Test No.	<i>p'010</i> (kPa)	$p'_i$ (kPa)	q (kPa)	OCR	SL	η(=q/ p' <sub>i</sub> )
(P <sub>010</sub> -NC-q670) <sub>shear</sub>	1000	1000	670	1	0.78	0.67
(P010-NC-q200) shear		1000	200	1	0.23	0.2
(P010-OCR1.5-q445) shear		666	445	1.5	0.78	0.67
(P010-OCR1.5-q200) shear		666	200	1.5	0.35	0.3
(P010-OCR4-q200) shear		250	200	4	0.93	0.8
(P <sub>010</sub> -NC-q670) <sub>creep</sub>	1000	1000	670	1	0.78	0.67
(P010-NC-q200) creep		1000	200	1	0.23	0.2
(P010-OCR1.5-q445) creep		666	445	1.5	0.78	0.67
(P <sub>010</sub> -OCR1.5-q200) creep		666	200	1.5	0.35	0.3
(P <sub>010</sub> -OCR4-q200) creep		250	200	4	0.93	0.8

# 703 Figure captions

- Fig. 1 Loading state of triaxial tests in (p' q) plane
- Fig. 2 Image processing method: (a) SEM image; (b) particle representation; (c) orientation
   curve; (d) binary image; (e) ellipses fitting; (f) schematic plot of the pore geometry
- 707 Fig. 3 Strain versus stress along monotonic loading for NC tests
- **Fig. 4** Evolution of creep deformation for NC tests: (a) creep strain (b) creep strain rate
- 709Fig. 5 Microfabric of tests  $P_{010}$ -NC-q670 in contractancy domain: (a) after shear (b) after710creep
- 711 Fig. 6 Strain versus stress along monotonic loading for tests OCR=1.5
- 712 **Fig. 7** Evolution of creep deformation for tests OCR=1.5: (a) creep strain (b) creep strain rate
- Fig. 8 SEM images of tests OCR1.5 after shear: (a) in pseudo-elastic domain (b) in contractancy domain
- Fig. 9 SEM images of tests OCR1.5 after creep: (a) in pseudo-elastic domain (b) in contractancy domain
- Fig. 10 Global particle orientation of specimens in NC tests: (a) after shear (b) q=200 kPa (c)
- 718 q=670 kPa (d) after creep
- Fig. 11 Global particle orientation of specimens in OCR=1.5 test: (a) after shear (b) q=200
  kPa (c) q=445 kPa (d) after creep
- 721 Fig. 12 Pore orientation versus stress level of NC tests: (a) after shear (b) after creep
- Fig. 13 Evolution of pore shape for NC tests after shear: (a) roundness (b) length (c) diameter
- Fig. 14 Evolution of pore shape for NC tests after creep: (a) roundness (b) length (c) diameter
- Fig. 15 Pore orientation versus stress level of tests OCR=1.5: (a) after shear (b) after creep
- Fig. 16 Evolution of pore shape for tests OCR=1.5: (a) roundness (b) length (c) diameter
- Fig. 17 Evolution of creep deformation for tests under stress level at  $\eta = 0.67$ : (a) creep strain (b) creep strain rate
- **Fig. 18** Global particle orientation of specimens under final stress level at  $\eta = 0.67$ : (a) after shear (b) after creep
- Fig. 19 Global pore orientation of specimens under final stress level at  $\eta$ = 0.67: (a) after shear (b) after creep
- Fig. 20 Evolution of pore shapes for creep samples under stress level at  $\eta$ = 0.67: (a) roundness (b) length (c) diameter
- **Fig. 21** Evolution of microstructure with *SL* of NC samples: (a) isotropic index (b) pore shape
- 735 Fig. 22 Illustration of microstructure evolution in different volumetric domains
- Fig. 23 Evolution of microstructure with *SL* of samples OCR=1.5: (a) isotropic index (b) pore
   shape
- 738 Fig. 24 Local coordinate at inter-particle contact
- 739 **Fig. 25** Fabric evolution of particles

















(b)











(P010-NC-q200)shear





P











































