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Characterisation of the multi-scale fabric features of high plasticity clays

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Abstract:	<p>The letter describes an investigation of the microstructural features of a high-plasticity clay, in both its natural conditions and reconstituted in the laboratory. Scanning electron microscopy is used to characterise the fabric; image processing of the micrographs delivers a quantitative assessment of the orientation of particles. Despite their identical composition, the natural and the reconstituted clay have experienced different deposition and loading history, generating different microstructural features that are shown to underlie their differences in state. For both clays, the fabric is shown to be well oriented at medium magnification. However, SEM observations at larger magnification, and the corresponding image processing results, reveal that areas of random particle aggregation alternate with zones with layers of perfectly oriented particles, providing evidence of multi-scale fabric features, which make fabric characterisation dependent on the scale of analysis.</p> <p>This peculiar fabric character is also identified in the two clays after 1D compression to high pressures, showing that, due to loading, fabric undergoes a complex re-organization but the index of fabric orientation remains largely constant. Thereby, it is concluded that electrostatic and short-range forces, rather than mechanical, are strongly influential during compression thus explaining the isotropic hardening observed in laboratory tests.</p>

Dear Editor,
please receive the original paper entitled:

“Characterisation of the multi-scale fabric features of high plasticity clays”

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for consideration for the themed issue of Géotechnique Letters “Latest Findings on Micro to Macro Mechanics of Geomaterials”.

The letter deals with the investigation of the microstructural features of high plasticity clays. Scanning electron microscopy and image processing are used for the qualitative and quantitative assessment of the clay fabric, while X-ray micro-analysis and swelling tests are adopted for estimating the bonding strength.

The microstructural analyses are carried out on both the natural clay and on the clay reconstituted in the laboratory, allowing to identify the microstructural differences underlying the differences in state. Moreover, the evolution of clay microstructure upon 1D compression of both the clays is investigated. Peculiar features of the clay fabric are outlined, providing evidence of its multi-scale architecture.

Looking forward to receiving from you a positive reply, we send our kindest regards.

Federica Cotecchia, Simona Guglielmi, Francesco Cafaro, Antonio Gens

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Abstract

The letter describes an investigation of the microstructural features of a high-plasticity clay, in both its natural conditions and reconstituted in the laboratory. Scanning electron microscopy is used to characterise the fabric; image processing of the micrographs delivers a quantitative assessment of the orientation of particles.

Despite their identical composition, the natural and the reconstituted clay have experienced different deposition and loading history, generating different microstructural features that are shown to underlie their differences in state. For both clays, the fabric is shown to be well oriented at medium magnification. However, SEM observations at larger magnification, and the corresponding image processing results, reveal that areas of random particle aggregation alternate with zones with layers of perfectly oriented particles, providing evidence of multi-scale fabric features, which make fabric characterisation dependent on the scale of analysis.

This peculiar fabric character is also identified in the two clays after 1D compression to high pressures, showing that, due to loading, fabric undergoes a complex re-organization but the index of fabric orientation remains largely constant. Thereby, it is concluded that electrostatic and short-range forces, rather than mechanical, are strongly influential during compression thus explaining the isotropic hardening observed in laboratory tests.

Keywords chosen from ICE Publishing list

clays; fabric/structure of soils; microscopy

List of notation

A	clay activity
CF	clay fraction
CRS	constant rate of strain oedometer test
C_s	swelling index
C_s^*/C_s	swell sensitivity
e	void ratio
L	index of fabric orientation
OCR	overconsolidation ratio: σ'_p/σ'_v
OED	conventional oedometer test
PI	plasticity index
S_σ	Stress Sensitivity
w	water content
YSR	yield stress ratio: σ'_y/σ'_v
σ^*_e	equivalent vertical effective stress on the ICL
σ'_p	vertical (geological) preconsolidation pressure
σ'_v	vertical effective stress

σ'_y vertical effective stress at yield

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1 1 **1. Introduction**

2 2 This letter discusses the features of the microstructure of a high plasticity clay in its natural
3 3 overconsolidated state, after one-dimensional (1D) compression in the laboratory (lab), and when
4 4 reconstituted and also subjected to 1D compression (Burland, 1990). In this way, the
5 5 microstructure corresponding to the different macro-states (void ratio, e , versus vertical effective
6 6 stress, σ'_v) achieved by the clay through either a geological or a lab history (Leroueil & Vaughan,
7 7 1990; Cotecchia & Chandler, 2000) is investigated. The micro-scale investigation has made use
8 8 of scanning electron microscopy (SEM), image processing of the micrographs at different
9 9 magnifications, X-ray micro-analysis (Energy Dispersive X-ray Spectroscopy, EDS in the SEM)
10 10 and, for the bonding strength, swelling tests.

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12 12 The presented work is part of a research aimed at connecting the features of clay macro-
13 13 behaviour (macro-mechanics) to the clay microstructure and history (e.g. Delage and Lefebvre,
14 14 1984; Hattab et al., 2013; Lima et al, 2008). Also, the research results are intended to support the
15 15 modelling of clays in the framework of micro-mechanics, that must account for complex coupled
16 16 chemo-physical processes at colloidal scale (e.g. Ebrahimi et al., 2012 and 2014, Anandarajah,
17 17 2000; Yao and Anandarajah, 2003, Ebrahimi et al., 2016; Liu et al., 2015; Sjoblom, 2016).

18 19 19 **2. Composition, history and macro-behaviour of the clay**

20 20 The investigated natural clay is the stiff Pappadai clay, deposited in the early Pleistocene in a
21 21 quiet marine environment, with reducing conditions at the sea floor. The mineralogical
22 22 composition and the index properties of the clay are reported in Table 1. It is mainly illitic, but
23 23 includes a significant amount of smectite, interstratified illite-smectite and carbonatic silt.

24 24 For the geological history of the natural clay and the macro-behaviour of both the natural and the
25 25 reconstituted clay, reference is made to Cotecchia & Chandler (1995 and 1997). A few aspects
26 26 of the clay macro-behaviour, which characterize the macro-effects of some clay micro-features,
27 27 are briefly recalled hereafter.

28 28 The state of the natural clay, A in the compression plane in Figure 1, results from
29 29 overconsolidation due to unloading ($OCR = \sigma'_p / \sigma'_{v0} = 3$). When subjected to oedometric

1 30 compression in the lab (Figure 1), the clay exhibits gross yield at σ'_y about twice σ'_p (yield stress
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3 31 ratio, $YSR = \sigma'_y / \sigma'_{v0} \cong 2 \cdot OCR$) as result of diagenesis under burial, which has increased the
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5 32 strength of the clay microstructure.

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7 33 Upon reconstitution and one-dimensional compression, the clay follows a compression curve to
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9 34 the left of the gross yield state of the natural clay (Figure 1), whose microstructure, achieved
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11 35 through the geological history, allows for $e - \sigma'_v$ states in the 'structure permitted space' (Leroueil
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13 36 & Vaughan, 1990).

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15 37 The swell sensitivity, i.e. C_s^* / C_{si} (Schmertmann, 1969), is about 2.5 and is indicative of a higher
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17 38 strength of the natural clay bonding with respect to that of the reconstituted, most likely due to
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19 39 diagenesis. For the undisturbed natural clay, the stress sensitivity S_σ , σ'_y / σ'^*_e ($\cong p'_{yis} / p'^*_{yis}$ in
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21 40 isotropic compression, Cotecchia and Chandler, 2000) equals 3.5 (Figure 1).

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23 41
24
25 42 Upon compression, the swell sensitivity of the natural clay drops to 1 soon after gross yield
26
27 43 ($C_s^* / C_{s,py}$, Figure 1), but S_σ decreases only gradually. Accordingly, the state of the natural clay
28
29 44 keeps lying to the right of the ICL up to high pressures. The clay microstructure is an internal
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31 45 variable of the hardening function in several constitutive laws (e.g. Rouainia & Wood, 2000;
32
33 46 Baudet & Stallebrass 2004). Cotecchia & Chandler (2000) proposed that S_σ , function of the plastic
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35 47 volumetric strain, ε_v^p , may be suited to represent the microstructure effects on the clay gross yield
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37 48 hardening. They support this proposal by means of experimental data for several clays, including
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39 49 Pappadai clay, showing that the gross yield surface of the tested materials can be normalized by
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41 50 the function $S_\sigma(\varepsilon_v^p) \cdot p_e^*(e)$ (where p_e^* is the equivalent pressure for the current void ratio of the
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43 51 reconstituted clay). Therefore, an isotropic volumetric hardening function, equal to the product of
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45 52 the current stress sensitivity of the clay times the hardening function of the reconstituted clay (e.g.
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47 53 the Cam Clay hardening function; Schofield & Wroth 1968), appears to match the gross yield
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49 54 hardening of Pappadai clay (Figure 2).

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53 56 **3. Microstructure of the natural and reconstituted clay**

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56 57 The analysis of the clay microstructure concerned vertical fractures of freeze-dried specimens,
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58 58 by means of SEM (gold coated) and Field Emission SEM (FESEM, carbon coated), as discussed
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1 59 by Cotecchia & Chandler (1998), Cotecchia et al. (2016) and Guglielmi et al. (2018). The
2 60 technique used for the digital image processing of the micrographs is operator-independent. It is
3
4 61 fully discussed by Martinez-Nistal et al. (1999) and Mitaritonna et al. (2014), and it is based on
5
6 62 the thinning of the elongated bright regions across the micrograph, which represent the edges of
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8 63 either particles, or particle aggregates (e.g. Figure 3). The thinning results in a field of vectors of
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10 64 varying orientation, processed to derive both a histogram of the detected particle orientations and
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12 65 a scalar “index of fabric orientation”, L (e.g. Figure 3). L ranges between 0.21 and 1 for “medium
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14 66 to very oriented fabric”, and is lower than 0.15 for “randomly oriented fabric”.
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16 67
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18 68 The reconstituted clay reaches state A^* in Figure 1 ($e=1.28$; $\sigma'_v=20\text{kPa}$) through compression
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20 69 from slurry in the consolidometer, up to $\sigma'_p=200\text{kPa}$, and swelling. When investigated at 10^3
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22 70 medium magnification (e.g. Figure 4), its fabric is found to match a repetitive pattern. This is
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24 71 formed of stacks, where densely packed domains are in either face to face, or edge to edge
25
26 72 contact, which alternate with either macro-pores, or arrangements of non-oriented
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28 73 particles/domains (mostly in edge to face contact). The repetitiveness of these fabric features
29
30 74 suggests that the medium magnification provides a representative view of the fabric on the whole.
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32 75 This fabric has been considered quite oriented by Cotecchia and Chandler (1997 and 1998) on a
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34 76 qualitative basis. Image processing, carried out recently for several medium magnification
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36 77 micrographs, confirms this identification, providing direction histograms like that in Figure 4, with
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38 78 L in the range 0.23-0.27.
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41 79 The medium magnification fabric of the overconsolidated natural clay, whose state is A ($e=0.88$;
42
43 80 $\sigma'_v=414\text{kPa}$) in Figure 1, appears repeatedly as a dense packing of massive aggregates (Figure
44
45 81 3). These are either domains arranged in stacks, or forming bookhouse arrangements, the latter
46
47 82 through edge to edge and edge to face contacts. Also chaotic particle aggregates, macropores
48
49 83 and unbroken micro-fossils can be found buried in the fabric. Cotecchia and Chandler (1997 and
50
51 84 1998) considered this fabric well oriented on qualitative basis. The image processing (e.g. overlay
52
53 85 in Figure 3a) confirms, again, this qualitative finding, providing direction histograms of the type in
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55 86 Figure 3, with L in the range 0.24-0.37. Therefore, irrespective of the different deposition
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57 87 environment, history and current state, both the natural and the reconstituted clay are shown to
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59 88 possess massive well oriented fabrics, once the analysis of the particle orientation concerns
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1 89 vertical fractures of size equal to, or larger than $10^4 \mu\text{m}^2$. It is deduced that, at the micro-scale, the
2 90 clay representative element volume, REV, to be investigated in order to assess the overall fabric
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4 91 features, is the cubic volume subtended by a surface of size $10^4 \mu\text{m}^2$, which corresponds to 10^{-3}
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6 92 mm^3 .
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8 93 A high degree of fabric orientation is achieved by the reconstituted clay already by $\sigma'_v=200\text{kPa}$.
9
10 94 The main differences between the fabrics at A and A* concern: i) the bonding, that is diagenized
11 95 in the natural clay, as indicated by the detection, through X-ray micro-analysis, of an amorphous
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13 96 calcite film binding the particles (Cotecchia & Chandler 1997); ii) the size and quantity of the
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15 97 macro-pores (μm) and the density of the domain aggregates; iii) the complexity of the clay fabric,
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17 98 which appears higher for the natural clay, including more frequent chaotic aggregations.
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23 100 When comparing the medium magnification micrographs for the reconstituted clay at A* with those
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25 101 for the reconstituted clay compressed to $\sigma'_v=22\text{MPa}$ (state C*, Figure 1), the difference in overall
26
27 102 fabric density is evident, given the absence of macro-pores and the thicker size of the massive
28
29 103 layers of oriented particles in C* (Figure 5). Conversely, a variation in fabric orientation cannot be
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31 104 detected. Image processing confirms that the average fabric orientation of the reconstituted clay
32
33 105 does not increase with the one-dimensional compression, since L keeps values about 0.24. A
34
35 106 similar finding was reported by Mitaritonna et al. (2014) for reconstituted Lucera clay, when one-
36
37 107 dimensionally compressed from $\sigma'_v=140\text{kPa}$ to 1900kPa . The authors also show that the
38
39 108 observed similarity in fabric orientation corresponds to a constancy in elastic stiffness anisotropy.
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41 109 In particular, different elastic stiffness anisotropies are shown to correspond to the different steady
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43 110 degrees of orientation that the clay achieves in different constant $\eta=q/p'$ compressions. Therefore,
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45 111 the authors suggest that the clay macro-behaviour at very small strains relates to the degree of
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47 112 orientation of the clay fabric detected at medium magnification, here recognized as the REV
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49 113 fabric.
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52 114 The role of the REV fabric as internal variable of the clay small strain macro-behaviour can be
53
54 115 extended to the large strain behaviour as shown in the following, based upon the reconstituted
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56 116 Pappadai clay data. As indicated above, the main change in REV fabric for the reconstituted
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58 117 Pappadai clay under one-dimensional compression (constant $\eta=0.6$) is the reduction in porosity,
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118 since the fabric orientation remains constant. This fabric evolution is therefore the internal process
 119 providing the reconstituted clay with a hardening function that is isotropic and volumetric, as
 120 suggested by the data in Figure 6, which show that the state boundary surface (SBS) of
 121 reconstituted Pappadai clay can be normalized by $p_e^*(=e^{(N^*-v)/\lambda^*})$.
 122 For the natural clay, compression to state B (Figure 1), soon after gross yield, is observed to
 123 cause a major weakening of the natural bonding, given the recorded drop in C_s^*/C_s . Figure 7
 124 shows one of several medium magnification micrographs for the clay at this state, which suggest
 125 that gross yielding results in some fabric rearrangement, including the chaotic filling of macro-
 126 pores with particle aggregates. However, the REV fabric does not attain a higher orientation
 127 degree, as confirmed by the image processing index L (Figure 7).
 128 With post-gross yield compression up to 25 MPa (state C), the natural REV fabric (Figure 8) is
 129 repeatedly formed by thickened stacks of face to face particles (probably result of the coalescence
 130 of original stacks), of extremely low porosity, interbedding chaotic fabric portions. The fabric is not
 131 significantly more oriented than before compression, as confirmed by the direction histogram in
 132 Figure 8 and the orientation index, $L=0.345$. Therefore, compression post-gross yield of the
 133 natural clay determines: i) a major reduction of the macro-pores and a probable reduction of the
 134 micro-pores within the aggregates (to be checked through mercury intrusion porosimetry); ii) the
 135 thickening of stacks, which are also distorted and include areas of turbulent particles/domains; iii)
 136 a minor increase of the REV fabric orientation. This recorded constancy in fabric orientation
 137 suggests that, also for the natural clay, the gross yield hardening may be function solely of the
 138 volumetric straining, as for the reconstituted clay. This appears to be confirmed by the
 139 normalization of the SBS of the natural clay through the function $S_{\sigma(\varepsilon_v^p)} \cdot p_e^*(v)$ in Figure 2
 140 (Cotecchia & Chandler 2000). For the natural clay, the hardening law must comply also with the
 141 evolving strength of the clay structure, through the function $S_{\sigma(\varepsilon_v^p)}$.
 142
 143 For all the investigated clay states, the inspection of micrographs at higher magnification than the
 144 medium one (i.e. 10^4 - 10^5) shows that the local fabric orientation varies from a complete preferred
 145 orientation (c.p.o.; Sides and Barden, 1970), to a poor orientation (bookhouse, cardhouse or
 146 honeycomb). For example, Figures 9a and b, for the initial states of the reconstituted and the
 147 natural clay respectively, and Figures 10a, 10b, 10c, for the reconstituted and the natural clay at

1 148 different stages of compression, show examples of fabric portions (10^{-6} mm³) where the degree
2 149 of orientation, L , drops below 0.21. Conversely, in the several c.p.o. fabric portions, L is found to
3
4 150 be very high. Therefore, the fabric orientation is not uniform and the local phenomena taking place
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6 151 under one-dimensional compression may be classified in two categories: a) the coalescence of
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8 152 domains bringing about the thickening and compaction of particle stacks (c.p.o.); b) the shift of
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10 153 domains and aggregates, to achieve a denser fabric packing, preserving the original features of
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12 154 edge to edge and face to edge fabric aggregates (e.g. Figs. 9 and 10). Hence, the external loading
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14 155 seems to generate mostly a mechanical displacement of particle aggregates, rather than the
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16 156 collapse of face to edge and edge to edge particle/domain arrangements that are controlled by
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18 157 electrostatic or short-range forces. This finding may be of interest in the micro-modelling of clay
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20 158 behaviour.

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24 160 **4. Conclusions**

27 161 The research findings reported here have highlighted the multi-scale nature of the clay fabric. The
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29 162 multi-scale analysis shows that, since the local features of the clay fabric detectable at high
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31 163 magnification are not uniform, it is necessary to characterize the REV of the micro-structure in
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33 164 order to assess the micro-scale source of the clay macro-response. For the clay under study, the
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35 165 REV size is 10^{-3} mm³, whose analysis requires image processing of micrographs at 10^3
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37 166 magnification.

39 167 The findings reveal the complexity of the fabric of the investigated multi-mineral clay, reconstituted
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41 168 in the laboratory, subjected to high consolidation stresses and diagenesis in its geological history.
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43 169 Despite the differences in fabric and bonding of the natural and reconstituted clay, though, the
44
45 170 REV fabric orientation is constant with constant η compression up to high pressures, and
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47 171 flocculated fabric portions do not necessarily undergo collapse due to external loading. Such
48
49 172 microstructural changes constitute the background of a volumetric isotropic gross yield hardening
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51 173 of both clays.

53 174 The multi-scale analysis of the natural clay fabric at very high pressures suggests that during
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55 175 compression, after a major weakening of bonding at gross yield, the micro-scale phenomena
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57 176 optimize the particle arrangement, which evolves into an alternation of slabs (c.p.o) and
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1 177 bookhouse fabric portions. Such fabric evolution allows the natural clay to keep void ratios higher
2 178 than those of the reconstituted clay, at any pressure.

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5
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7
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14 184 processing of the micrographs.

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283 Table 1. Index properties, mineralogy and initial state of natural Pappadai clay (after Cotecchia
 284 and Chandler, 1997).

Composition and physical properties	Specific gravity, G_s	2.75
	Clay fraction, CF	58%
	Liquid limit, LL	65%
	Plasticity index, PI	35%
	Activity, A	0.6
	Natural water content, w_0	≈31%
	In situ void ratio, e_0	0.88
	Carbonate content	28%
Mineralogy	Quartz	3%
	Feldspar	1%
	Carbonate	22%
	Dolomite	6%
	Kaolinite	12%
	Chlorite	14%
	Illite	20%
	Smectite	12%
	Interstratified	10%
Total	100%	

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Figure 1

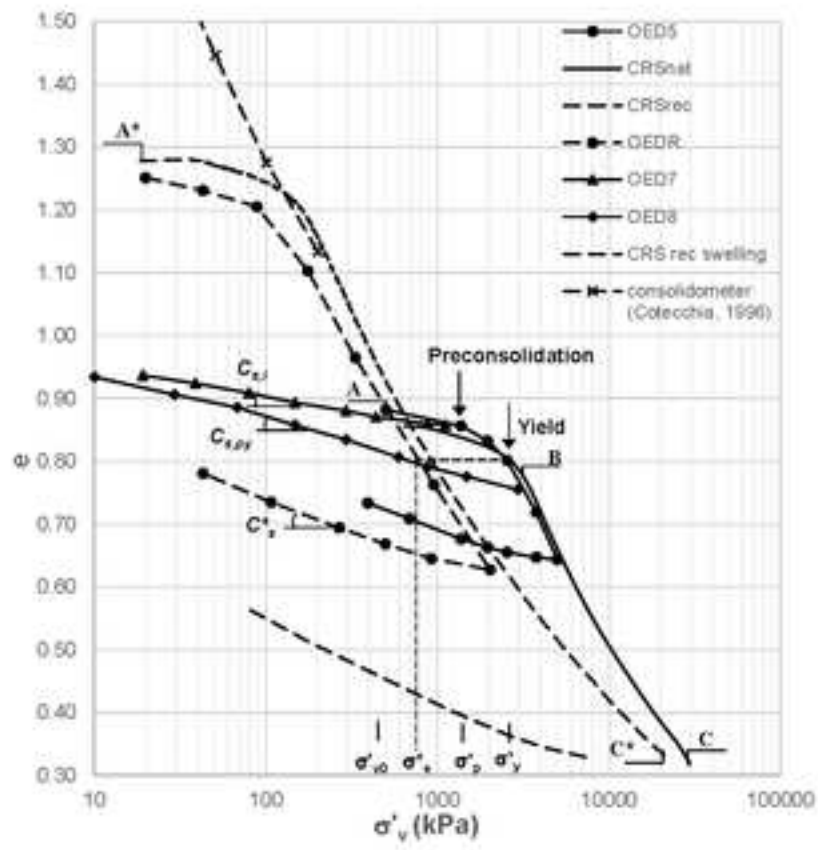


Figure 2

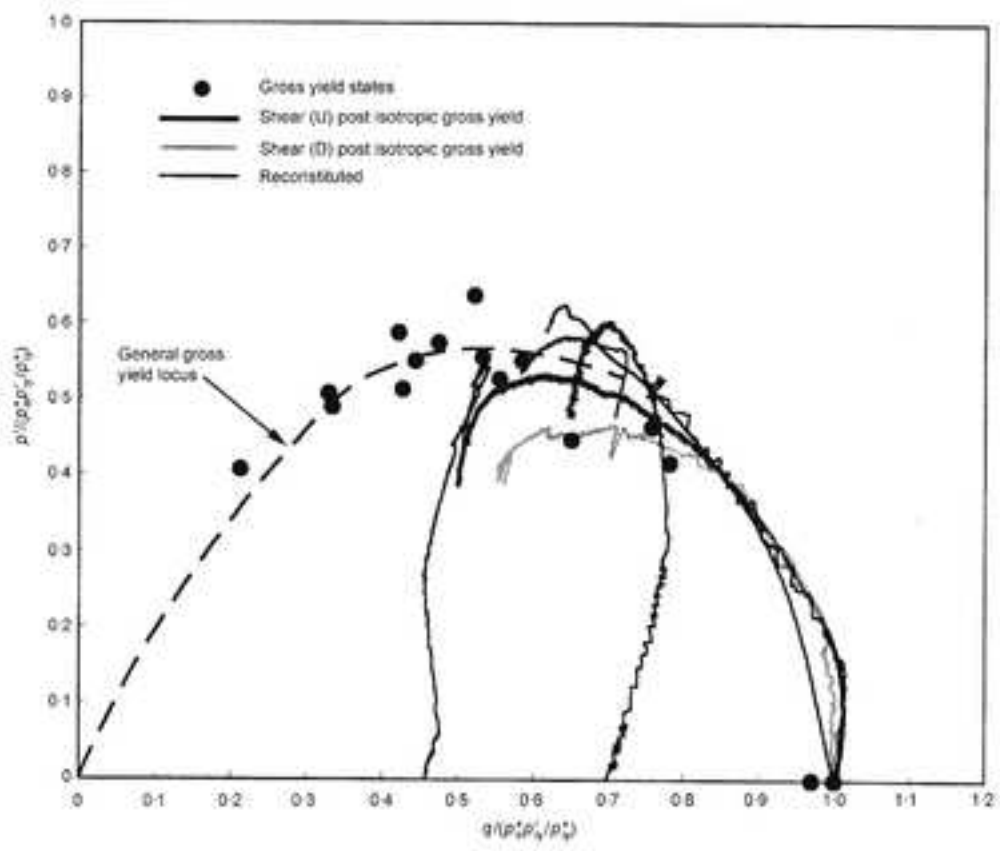
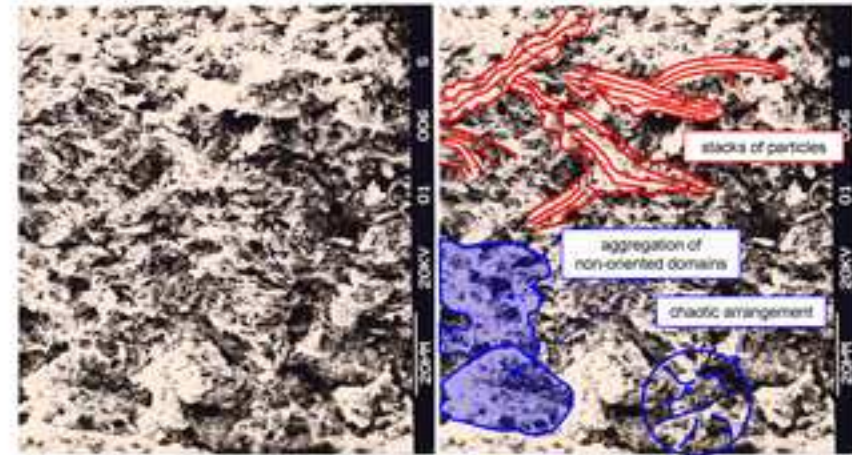
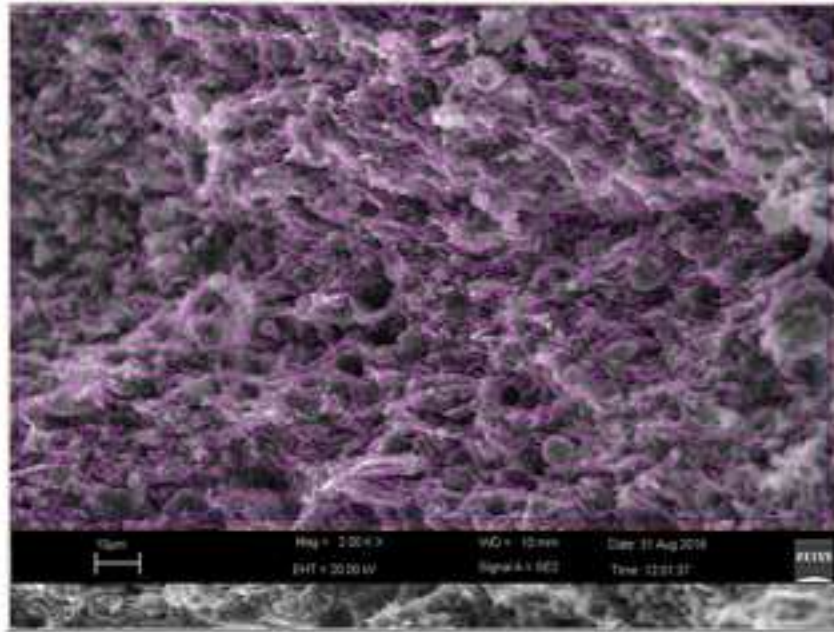
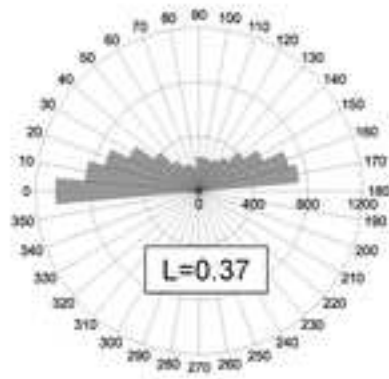


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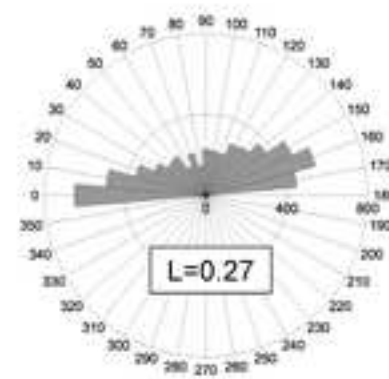


i)

ii)

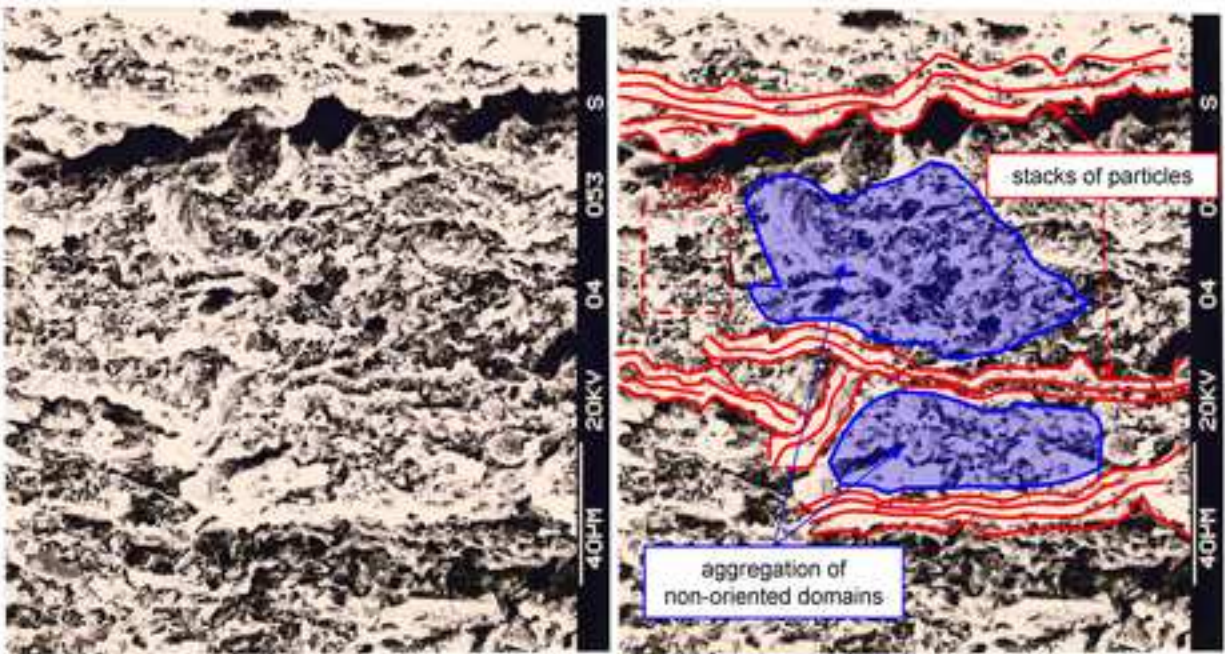


a)



b)

Figure 4



i)

ii)

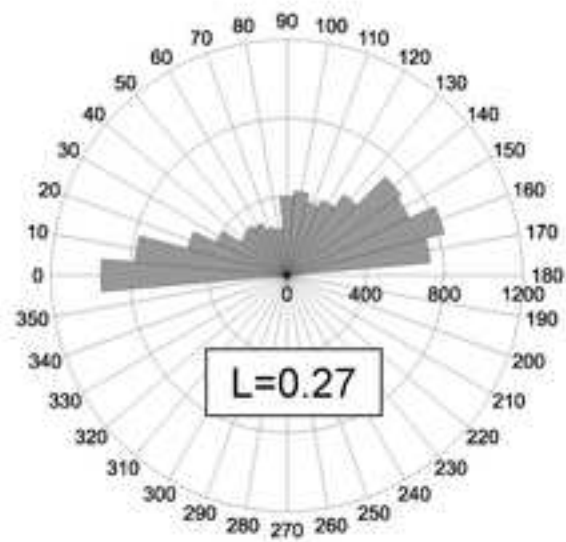


Figure 5

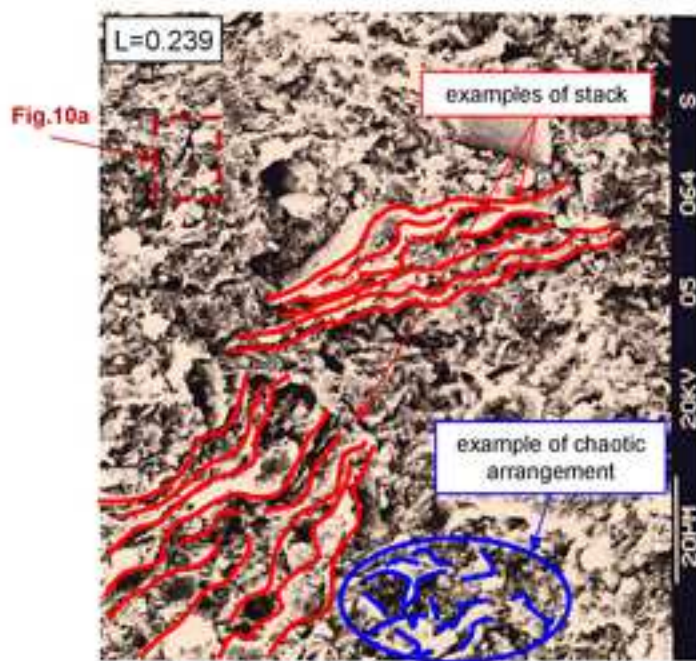


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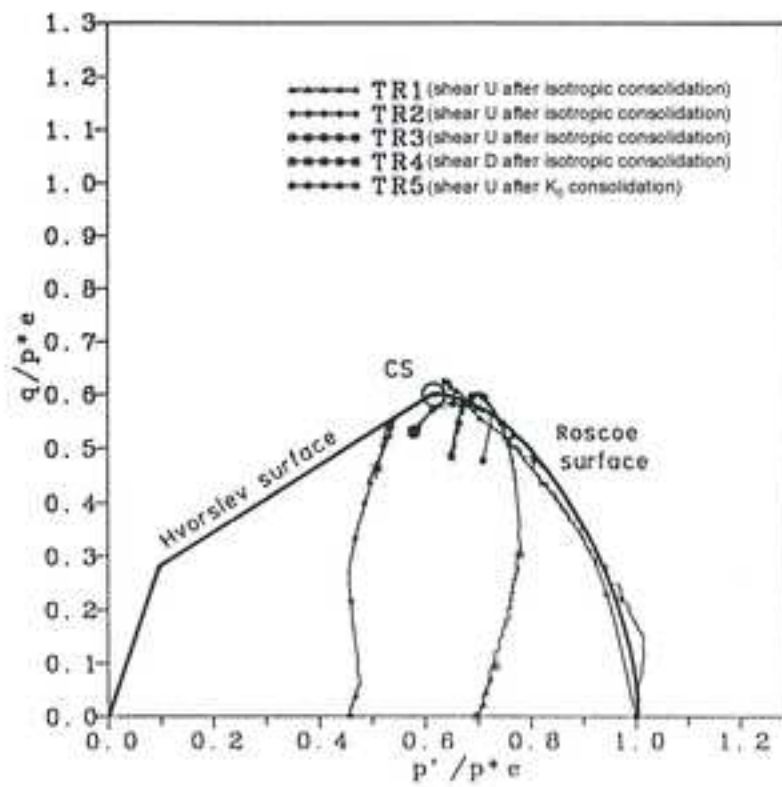


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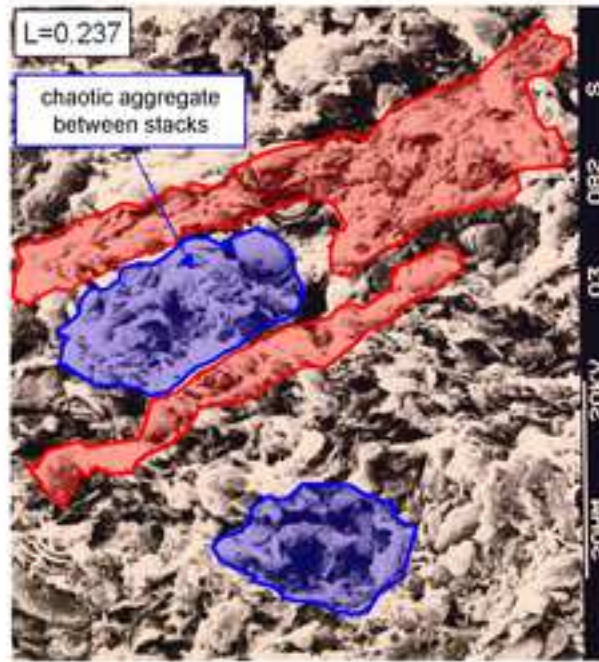


Figure 8

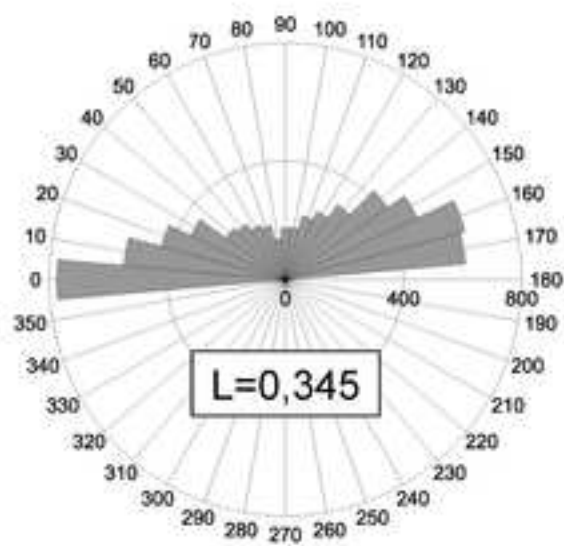
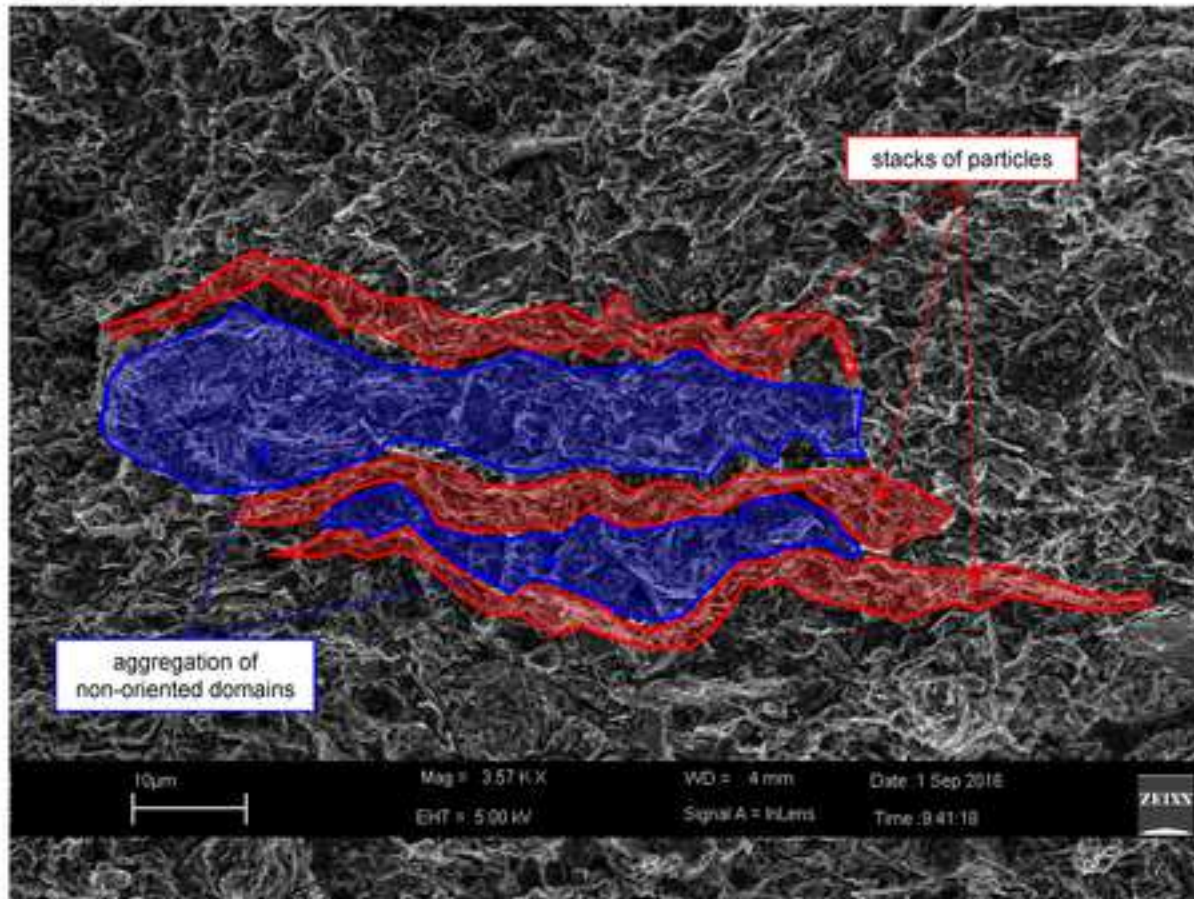


Figure 9



Figure 10

