# Variation in geometric arrangement of particles in kaolinite clay due to shear deformation using SEM technique

**Ajanta Sachan**

Department of Civil Engineering, Indian Institute of Technology, Kanpur 208 016, India

**Geometric arrangement of particles in a soil mass reflects the imprints of the geologic and stress history of the deposit, the depositional environment, and weathering history. Therefore, it is believed that the arrangement of particles in soils has a profound impact on its mechanical properties, such as shear strength and modulus. The present article deals with the impact of shear deformation on the geometric arrangement of particles within the specimen of kaolinite clay using high resolution SEM images. Specimens with two extreme geometric arrangements of particles have been prepared using slurry consolidation technique with and without dispersing agent: (i) specimen A (random geometric arrangement of particles), and (ii) specimen B (preferred geometric arrangement of particles) to evaluate the change in geometric arrangement of particles due to shear deformation with respect to the original one. Specimen B exhibited larger variation in geometric arrangement of particles due to shear deformation in comparison to specimen A. Thus the geometric arrangement of particles must be considered for predicting the shear strength behaviour of the soil accurately. Unfortunately, this has not been considered in most of the constitutive models used in practice for solving geotechnical engineering problems.**

**Keywords:** Clay, particle orientation, shear deformation, triaxial.

IN soil mechanics, considerable effort has been devoted to the determination of mechanical properties of the soil such as strength, compressibility and permeability. It is evident that all such factors depend, among other things, upon the geometric arrangement of particles in soil mass. Hence, a study on the geometric arrangement of particles and contact forces between them is needed to understand the shear behaviour of the soil at micro as well as macro levels. Geometric arrangement of particles in clay suspensions can be described in the form of random particle orientation and preferred particle orientation. Random orientation refers to clay particles of a sample that are oriented in all possible directions with equal probability, whereas preferred orientation refers to particles that are aligned parallel to each other.

When a soil specimen is subjected to external loads, localized deformation regions in the form of shear bands at failure are developed. McKyes and Yong[1] reported that the clay particles in the proximity of observed shear bands at failure align themselves along the failure plane. If we can assume this to be true for other cohesive materials as well, then based on the observations from the present study, the failure condition at the macro level may be derived using a correlation between the inter-particle forces at micro level and the void ratio of the soil at macro level based on the geometric arrangement of the particle. However, the shear behaviour of clay at micro level and the mechanics of clay particles and the inter-particle interactions are largely unknown; and this was an inspiration for the present work to study the variation in geometric arrangement of particles before and after shear deformation of the clay mass using scanning electron microscope (SEM) technique. This will provide knowledge and direction to this area of research using the latest testing facilities such as those used for studying particulate mechanics. Due to time constraints, only drained compression tests have been performed in the present study for two types of geometric arrangements of clay particles: random and preferred. Such a study would be able to answer questions related to the onset and propagation of localized deformations in soils under shear strength testing conditions, which would affect the analysis of shear strength parameters of cohesive soils (these parameters are used for designing foundations for civil engineering structures).

## Previous investigations

Many previous researchers[2–7] used a variety of techniques for the study of particle orientation of clayey soils using X-ray diffraction, SEM and transmission electron microscope (TEM). Most of the earlier studies[8–12] concentrated on the variation in particle orientation of clay due to compaction and consolidation of soil. McKyes and Yong[1] analysed the particle orientation of clay that experienced shear distortion using SEM images of the specimen at very low magnification (×10 and ×100). This did not provide

e-mail: ajantas@iitk.ac.in

any conclusive change in the particle orientation of the soil specimen due to the shear deformation process. The present study provides a complete analysis of geometric arrangement of particles in kaolinite clay focusing on the orientation of particles, formation of domains and clusters, and their geometric arrangement within the soil mass due to shear deformation by obtaining SEM images at high resolution (×2000 to ×40,000). In the present study, slurry consolidation technique was used to prepare two extreme geometric arrangements of particles for kaolinite clay[13]: (i) specimen A (without using dispersing agent, random geometric arrangement of particles), and (ii) specimen B (using dispersing agent, preferred geometric arrangement of particles). After slurry consolidation, specimen A was found to have predominantly face-to-edge contact and specimen B with predominantly face-to-face contacts of particles, as shown in Figure 1 at the resolution of 14,000.

## Material properties

Kaolinite clay has been chosen for this study because of the following reasons: (i) This is a stable clay mineral consisting of only one silica and one alumina sheet firmly bonded to each other. (ii) It is widely distributed in nature and usually the dominant clay mineral because of its relative resistance to chemical weathering. Due to the stable nature of this clay, it does not swell or expand in the presence of water. Thus the variation in geometric arrangement of particles could be observed only due to the shear deformation of soil specimens without being influenced by other effects.

All the experiments were performed on a commercially available pure kaolinite clay obtained from Akrochem Corporation, Akron, Ohio, under the trade name 'Akrochem SC-25'. The kaolinite clay used in this study had a liquid limit of 62%, plasticity index of 30% and specific gravity of 2.63. Grain-size distribution of kaolinite clay indicated that 92% of particles were finer than 10 μm, and 62% were finer than 2 μm.

## Crystal structure of kaolinite clay

The present study concentrates on geometric arrangement of particles in kaolinite clay. Therefore, it is necessary to know the kaolinite clay crystal structure, atoms placed inside a crystal unit, and arrangement of crystal units within a clay particle. The structure of kaolinite clay con-

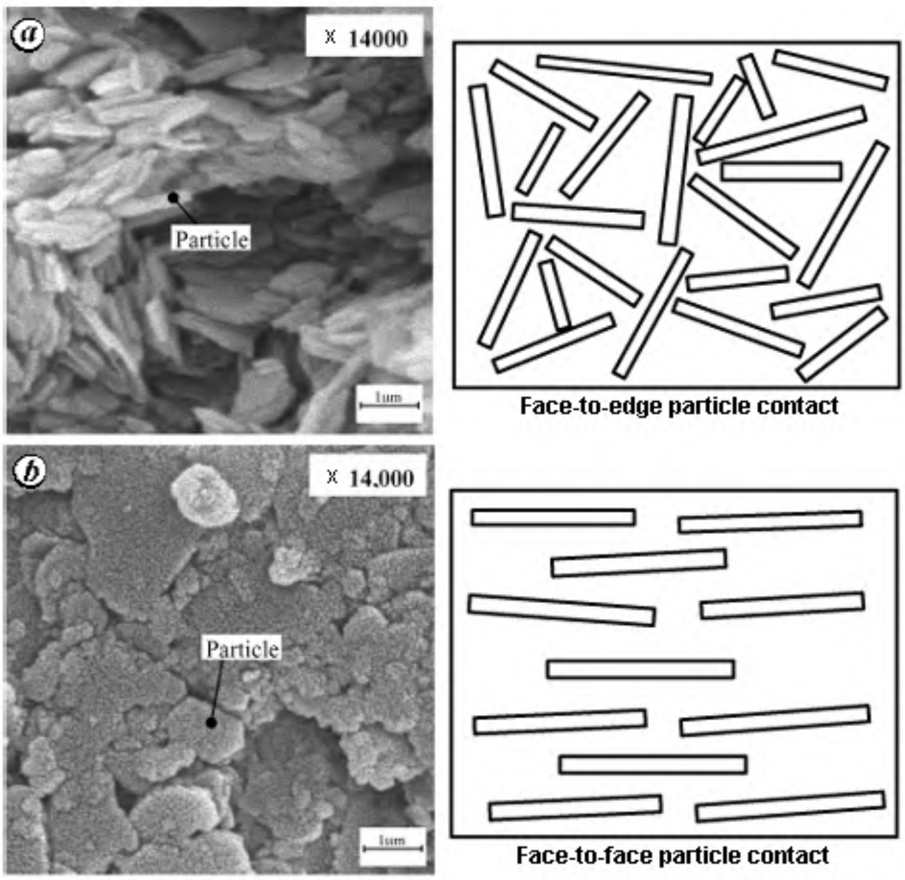


**Figure 1.** SEM image of kaolinite clay specimens before shear deformation. ***a***, Specimen A (random orientation, face-to-edge particle contact). ***b***, Specimen B (preferred orientation, face-to-face particle contact).

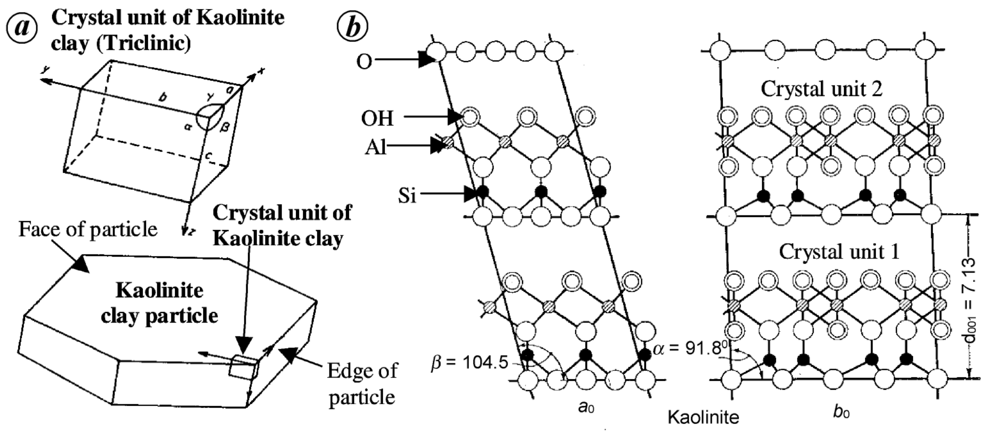


**Figure 2.** Particle and crystal unit of kaolinite clay. ***a***, Crystal unit and particle (after Carroll[15]); ***b***, Arrangement of atoms in the crystal unit (Brindley[2]).

sists of tetrahedral sheets of $SiO_2$ (silica) bonded to octahedral sheets of $Al_2O_3$ (alumina). Silica and alumina sheets are continuous in the *a* and *b* directions and stacked one above the other along the *c* axis. Yong and Hewat[14] reported that the kaolinite clay mineral has a triclinic structure. As shown in Figure 2, the specifications of a crystal unit for kaolinite clay are: $a = 0.515$ nm, $b = 0.895$ nm, $c = 0.715$ nm, $\alpha = 91.8°$, $\beta = 104.8°$ and $\gamma = 90.0°$. Each atomic layer has a thickness of about 7.2 Å (tetrahedral sheet = 2.1 Å and octahedral sheet = 5.1 Å). Figure 2 *b* shows the arrangement of atoms (Al, Si, O, OH) in a crystal unit of kaolinite clay and the arrangement of two units within a particle of kaolinite clay. Carroll[15] reported that approximately 600 units form one particle of kaolinite, which is about 9.0 nm wide, 10.3 nm long and 2.1 nm thick ($10 \times 20 \times 3$ cells). Several particles make one domain and several domains make one cluster. It is important to note that the number of particles within a domain or the number of domains within a cluster entirely depends upon the stress history and loading/boundary conditions of the clay mass.

## Specimen preparation

In the present study, a series of drained compression triaxial tests have been performed on solid cylindrical specimens of kaolinite clay with random (specimen A) and preferred (specimen B) particle orientation to evaluate the change in geometric arrangement of particles before and after shear deformation of the soil. This would provide a clear understanding of the shear bands and failure plane observed during shearing. These specimens were prepared in the laboratory using slurry consolidation technique. In this technique, the slurry was prepared with 155% water content corresponding to 2.5 times its liquid limit, as suggested by Sheeran and Krizek[16], to avoid the influence of the method of placement in a consolidometer. Kaolinite clay specimens with different particle orientations (preferred and random) were prepared with and without dispersing agent respectively. Two per cent of Calgon (dispersing agent) was used for preparing the clay slurry to obtain the kaolinite clay specimen with preferred particle orientation (specimen B) without changing the chemical composition of the soil[13]. An axial stress of 207 kPa was applied to the clay slurry during its $K_0$ consolidation for obtaining uniform and homogenous specimens of kaolinite clay. It is important to note that if the applied axial stress is less than 207 kPa, handling of the specimen (from slurry stage to solid cylindrical stage) becomes difficult[17]; and if the axial stress is more than 207 kPa, the effect of 1D consolidation becomes prominent during triaxial testing.

A series of isotropically consolidated drained triaxial tests have been performed on these solid cylindrical clay specimens (specimens A and B). The experimental process involved isotropic consolidation at 276 kPa and then shear deformation at a chosen axial strain rate. Specimen A was sheared at 0.005% per min and specimen B at 0.002% per min to allow dissipation of excess pore pressure during application of anisotropic loading under drained testing conditions. These strain rates were calculated for both types of geometric arrangements of particles (preferred and random) using consolidation-time data obtained during isotropic consolidation of the specimen[13]. It is important to note that the parallel-type particle orientation (specimen B) would take more time for dissipating excess pore pressure during consolidation as well as shear deformation in comparison to the random-type particle orientation (specimen A), as shown in Figure 3. Thus, much slower strain rate would be required for specimen B than for specimen A, as suggested by Sachan and Penumadu[13]. Lubricated end platens were used throughout the study in the triaxial testing system to avoid non-uniformity of stress state and deformation mode along the height of a specimen during the application of anisotropic loading. In the present study all possible disturbances and effects were tried to reduce/avoid as much as possible in order to evaluate the change in geometric arrangement of particles only due to shear deformation of the clay specimen. After the triaxial test, the cylindrical clay specimens were carefully removed from the assembly and then smaller, rectangular-shaped samples (10 × 10 × 2 cubic mm) for SEM analysis were obtained from the deformed cylindrical specimen. The 'unavoidable disturbance' of the specimen surface during the trimming process was minimized by peeling-off the surface layers using double stick tape.

## Discussion

Clay particle sizes are in the micrometre range length scale. The small dimensions of clay particles have a major influence on the molecular scale behaviour and interactions (particle–particle, particle–water and interlayer) of bulk mechanical properties. The basic structural units in clay consist of the silica sheets formed by tetrahedral silica and octahedral units formed by octahedrally coordinated cations (with oxygen or hydroxyl ions)[4]. Kaolinite mineral is made up of many micelles piled one on top of another. Since the surface of one micelle contains hydrogen ions and the other surface only oxygen ions, there is a tendency for hydrogen bonds to form between micelles. While individual hydrogen bonds are of low energy, the bonding energy is additive and the sum of the many hydrogen bonds between micelles results in the micelles being strongly bonded together and nearly impossible to separate. This bonding of the layers together results in kaolinite being a stable clay mineral. Particle orientation and its association with other particles within the clay mass can be described in the form of two types of particle orientations: face-to-edge and face-to-face particle contacts. This concept is the same for clusters as well as domains.

The mechanism of clay concentration and orientation within the matrix results in a structure that may act like one large crystal, although it may be composed of hundreds or thousands of individual clay particles. These particles have a platelet-like structure, which has an average diameter of 2 μm. In soils that have applied stress, release of internal stress may result in the rearrangement of clay particles or particle groups producing oriented clay along shear planes, around stable structural units, or within the soil matrix. In these features there has been no clay particle movement, only *in situ* rearrangement of clay domains or clay clusters (domain: group of particles; cluster: group of domains). The *in situ* rearrangement of clay particles due to shear deformation of clay, formation of domains, relationship between domains and clusters, and geometric arrangement of clusters within the soil mass for specimens A and B will be discussed in the following sections.

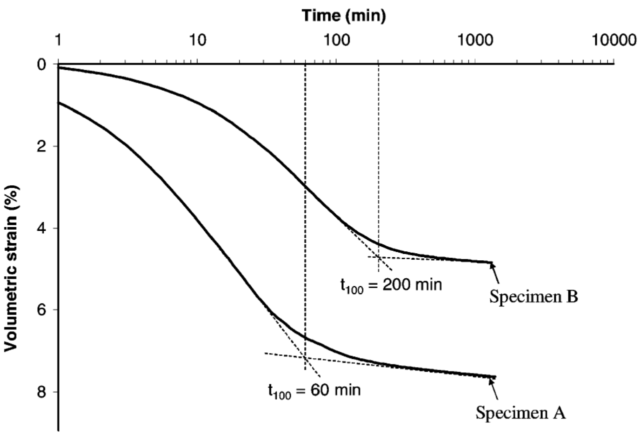


**Figure 3.** Volumetric strain versus time (log scale) for specimens A and B at 276 kPa of consolidation pressure.

### *Specimen A (random geometric arrangement)*

SEM image of kaolinite clay specimen A (Figure 1 *a*) showed face-to-edge contact between the particles, and no formation of domains (a group of particles which act as a unit) and clusters (a group of domains which act as a unit) within the clay mass before shear deformation. Since the clay specimens used in the present study were slurry consolidated in the laboratory and did not experience aging (time-dependent structural readjustment[18]) before triaxial testing; therefore, cohesion between particles was negligible before shear deformation. Thus, domains and clusters were not observed in the SEM image obtained before shearing (Figure 1). However, after shear deformation the SEM image of specimen A (Figure 4) exhibited face-to-face contact between particles and also showed the formation of domains and clusters within the clay mass. In the present study, the slurry consolidation technique allowed the clay slurry (water content = 155%) to let water drain out in order to obtain a solid cylindrical clay specimen (water content = 43.5%, void ratio = 1.13). This was later sheared under compression-drained triaxial testing conditions, resulting in the water content to be 30.4% (void ratio = 0.79), as listed in Table 1. Thus, reduction in void space between particles compelled them to move towards each other, resulting in the formation of the domain (a group of particles acting as a unit), and then the formation of the cluster (a group of domains acting as a unit). Figure 4 shows that the contact between domains is face-to-face, and that between clusters is face-to-edge. When the specimen was subjected to external anisotropic loading in compression triaxial shearing, the clay particles came closer to each other at an early stage of shear deformation (Figure 5) and formed a parallel orientation (face-to-face contact between particles). This resulted in the formation of clay domains such that they also acquired a platy shape and acted as unit. At high stress levels, clay particles might not be able to move due to the lack of enough free space (smaller void space) among the particles. This would lead to the parallel orientation of platy-shaped do-

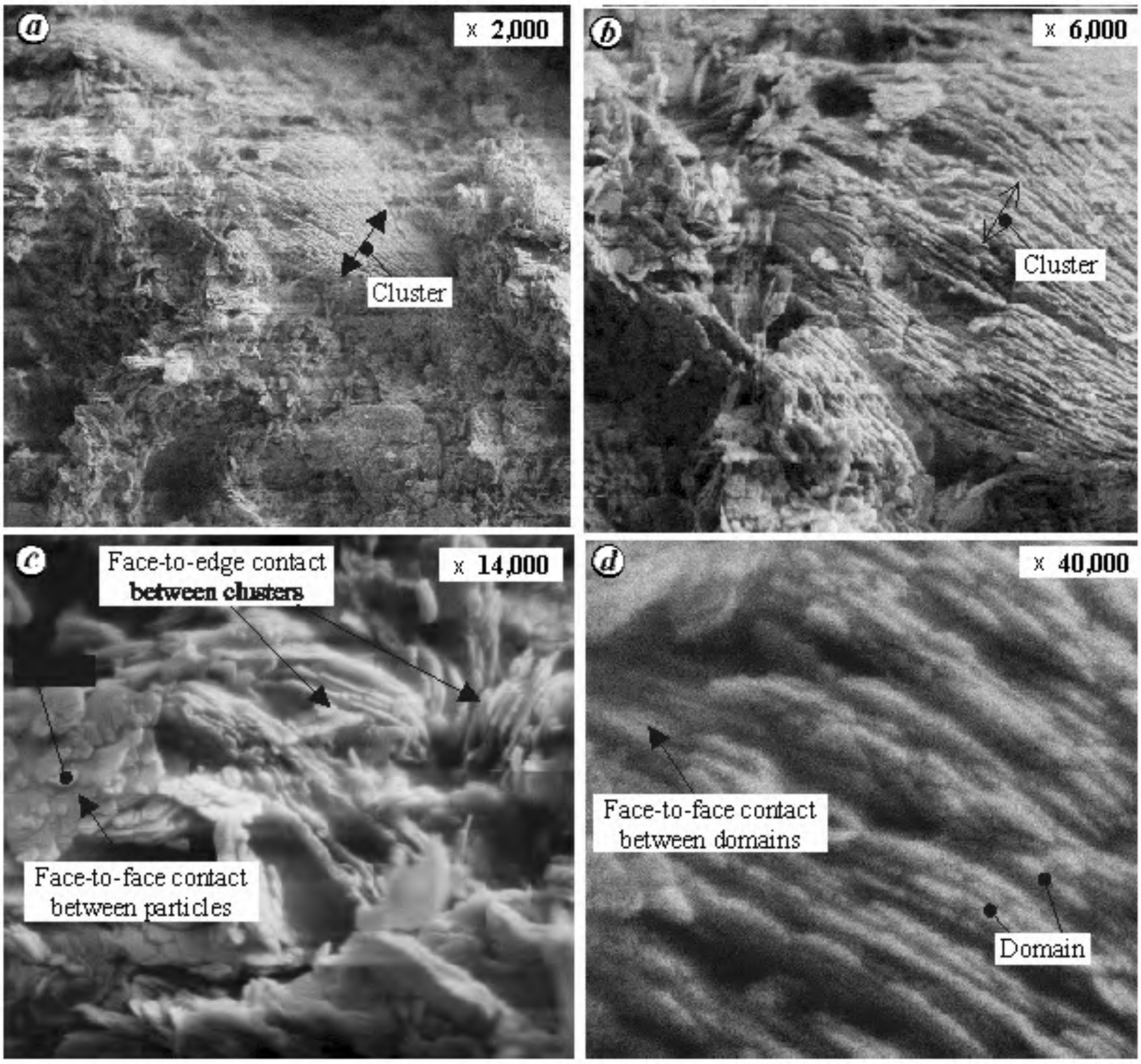


**Figure 4.** SEM images of kaolinite clay specimen A after shear deformation.

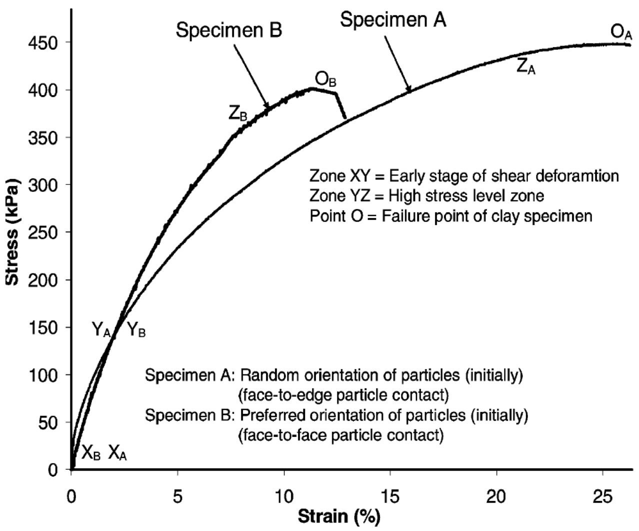


**Figure 5.** Stress–strain behaviour of kaolinite clay for specimens A and B.

mains, causing the formation of clusters within the clay mass. On further shearing (near to failure), these clusters would try to form a stable geometric arrangement within the specimen. Thus, they would orient themselves according to the direction of the shearing plane. This resulted into the face-to-edge contact between the clusters in specimen A, as shown in Figure 4 *c*. It is important to note that the clay particles are platy in shape, and this enhances the tendency of particles to orient parallel to each other within a domain under applied axial compression loading. Therefore, contact between clay particles was observed to be face-to-edge before shear deformation and face-to-face after shear deformation in SEM images of kaolinite clay specimen A.

### *Specimen B (preferred geometric arrangement)*

Unlike specimen A, contact between particles in specimen B was observed to be the same (face-to-face) before and

**Table 1.** Variation in geometric arrangement of particles, domains and clusters before and after shear deformation of kaolinite clay

| | Specimen preparation | Before shear deformation | After shear deformation |
|---|---|---|---|
| Specimen A (type of contact between two particles/domains/clusters) | Water content ($w$) of slurry = 155%. No dispersing agent used in clay slurry | $w$ = 43.5%, $e$ = 1.13<br>Particles: face-to-edge<br>Domains: do not exist<br>Clusters: do not exist | $w$ = 30.4%, $e$ = 0.79<br>Particles: face-to-face<br>Domains: face-to-edge<br>Clusters: face-to-edge |
| Specimen B (type of contact between two particles/domains/clusters) | Water content ($w$) of slurry = 155%. Two per cent dispersing agent used in slurry | $w$ = 29.0%, $e$ = 0.72<br>Particles: face-to-face<br>Domains: do not exist<br>Clusters: do not exist | $w$ = 23.5%, $e$ = 0.58<br>Particles: face-to-face<br>Domains: face-to-edge<br>Clusters: face-to-edge |

Void space (space between clay particles) is proportional to void ratio ($e$) and water content ($w$).

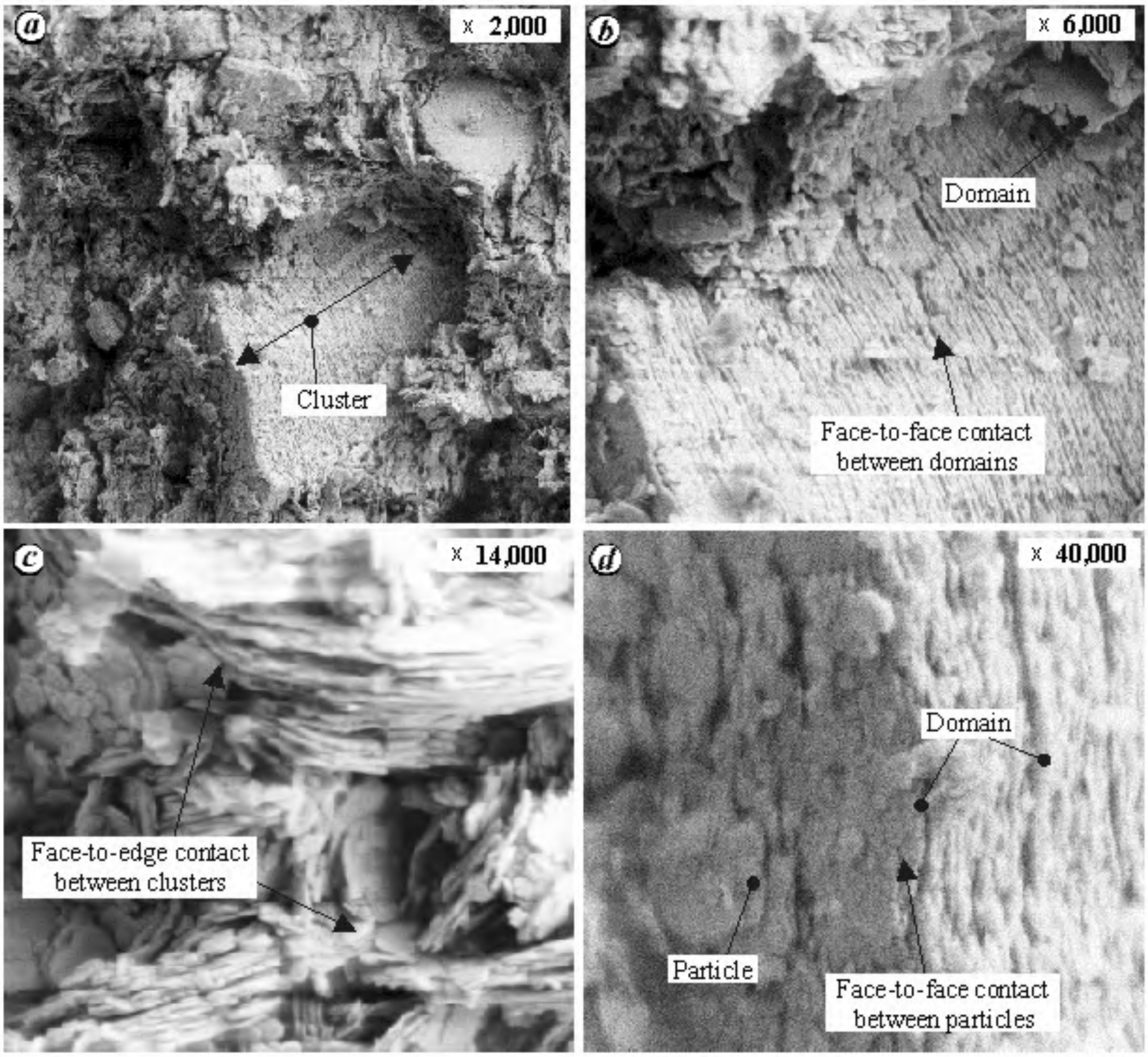


**Figure 6.** SEM images of kaolinite clay specimen B after shear deformation.

after shear deformation. When the specimen was subjected to anisotropic compression loading, particles were forced to come closer to each other and orient themselves parallel to each other based on the direction of loading and the initial orientation of the platy-shaped clay particle, which has a natural tendency to lie down on its face. Thus, the application of compression loading stimulated the formation of face-to-face contacts between the particles in specimen B (Figure 6). Similar to specimen A, domains and clusters were also not observed before shear deformation in specimen B due to the absence of aging effect (cohesion) in clay.

The size of domains and clusters was observed to be much larger in specimen B than in specimen A (Figures 4 and 6). The reason for this is due to the variation in initial geometric arrangement of particles in specimens A (face-to-edge particle contact) and B (face-to-face particle contact) before shear deformation. Since particles in specimen B were already arranged in face-to-face contact, application of compression loading helped the particles maintain their face-to-face contact, resulting in the formation of domains and then clusters at an early stage of shearing. These domains and clusters expand in size on further shearing (near to failure) leading to the formation of larger sized domains and clusters in specimen B compared to specimen A.

It is important to note that the boundary conditions (lubricated ends), loading conditions (compression), specimen stress history (normally consolidated), drainage conditions (drained test) in the triaxial tests performed on specimens A and B have been kept the same. In specimen B (preferred geometric arrangement), the parallel stacking of clay platelets has the capacity of carrying larger amount of axial load applied perpendicular to the faces of the platelets. However, at the same time platelets tend to slip suddenly at high stress levels. The slipping of platelets within specimen B leads to a loose configuration of platelets in a local zone induced by its less contractive nature, which has been observed to be the opposite for specimen A (denser configuration of platelets). These weak zones expand in size and propagate towards each other on further shearing. In this process, when several of these weak zones are connected, they may lead to significantly localized failure response. The orientation of clay platelets and distribution of local strains in these weak zones at high stress levels have been studied using digital imaging technique[19]. The distribution of local strains in these weak zones controls the orientation of domains and clusters within the soil specimen with respect to the geometric arrangement of particles before shear deformation.

## Conclusion

Using the SEM technique, the geometric arrangement of particles within the kaolinite clay specimens was studied before and after shear deformation under drained triaxial compression testing conditions. These experiments were performed on slurry consolidated specimens with two types of initial particle orientation: random (specimen A, predominantly face-to-edge particle contact) and preferred (specimen B, predominantly face-to-face particle contact). Key observations from this study are summarized below:

*Specimen A (random particle orientation initially)*

- The contact between clay particles was observed to be face-to-edge before shear deformation of kaolinite clay specimen A. However, it was observed to be face-to-face after shear deformation due to the large reduction in void space between the particles subjected to anisotropic compression loading during deformation.
- Domains and clusters were observed only after shear deformation (and not before) because clay specimens used in the present study were slurry consolidated in the laboratory and did not experience aging, resulting in no cohesion between the particles before shear deformation.

*Specimen B (preferred particle orientation initially)*

- Unlike specimen A, the contacts between particles were observed to be the same (face-to-face) before and after shear deformation in specimen B due to the direction of loading and natural tendency of platy-shaped clay particles.
- The size of the clusters was observed to be larger in specimen B than in specimen A due to the initial orientation of particles (face-to-face contact) in the former.
- Domains and clusters were also not observed before shear deformation of specimen B due to the similar reason as explained for specimen A.


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# MEETINGS/SYMPOSIA/SEMINARS

**8th Indo-US Cytometry Workshop—Clinical Applications of Flow Cytometry**

Date: 19–22 December 2007
Place: Lucknow

The workshop will teach the fundamentals of flow cytometry for beginners and give special emphasis for its use in diagnosis and management of various clinical conditions like leukaemias, lymphomas, idiopathic thrombocytopenic purpura, pre-transplant cross match, analysis of fluids in solid tumors, apoptosis, etc.

Contact: Workshop Secretariat,
Department of Immunology,
Sanjay Gandhi Postgraduate Institute of Medical Sciences,
Raebareli Road,
Lucknow 226 014
Phone: 91-522-2668700/800/900 Ext 2284
E-mail: cytometry2007@sgpgi.ac.in
Website: www.cytometry2007.com

**GEOTRACES Indian Ocean Planning Workshop**

Date: 24–26 October 2007
Place: Goa

The goal of the workshop is to refine the scientific objectives developed in the GEOTRACES science plan (available from www.geotraces.org), and place those objectives into a framework of Indian Ocean sections and process studies. An important aspect of the workshop is to identify nations that are prepared to take the lead in carrying out specific cruises as elements of the broader basin-scale plan. Individuals who are interested in participating in GEOTRACES programme and/or who are prepared to organize research cruises and to secure funding to support those cruises are particularly encouraged to participate.

If you wish to participate in the workshop and know more about it please visit www.prl.res.in/~geotraces-india

**National Conference on Latest Trends in Nanotechnology**

Date: 5 January 2008
Place: Thane

Topics include: Processing and fabrication of nanomaterials; Characterization of nanomaterials; Nanodevicer; Applications of nanotechnology; Potential risks associated with the technology.

Contact: Mrs Usha Raghavan
Head, Department of Information Technology,
VPM's Polytechnic,
'Jnanadweepa', Chendai Bunder Road,
Thane College Campus,
Thane (W) 400 601
Phone: 022-25446551, 022-25364494
E-mail: nanovpm@gmail.com
Website: www.vpmthane.org