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Mechanical and chemical properties for self-healing cement-treated clay

Propriétés mécaniques et chimiques de l'argile auto-cicatrisante traitée au ciment

Kiyonobu Kasama & Tomoro Ueda
Civil and Environmental Engineering, Tokyo Institute of Technology, Japan, kasama.k.ab@m.titech.ac.jp

ABSTRACT: In order to develop a self-healing ground improvement technology, the recovery characteristics of the shear strength for cement-treated Kawasaki and Kaolin clays was clarified by means of a series of box shear tests and powder X-ray diffraction analysis. The main conclusions are as follows: 1) The shear strength recovery depends on the soil type of base material, GGBS content and the vertical pressure for box shear test. Cement-treated Kawasaki clay shows a strong shear strength recovery as the range from 7.4 % to 340.8 %. 2) Four indexes were proposed to express the shear strength recovery for cement-treated clay. GGBS is effective for increasing the initial shear strength, however, GGBS has negative effect on the strength recovery of cement-treated clay. 3) The recovery shear strength consists of a large friction component although conventional cement-treated clay has a large cohesion component. It can be considered that the cement chemical reaction on shear surface after shearing augments the fictional resistance instead of cohesive component. 4) The shear strength recovery of self-healing cement-treated clay is considered to be due to the increase of Ettringite and Calcium Aluminate Hydrate by the hydration of Unhydrate cement on the shear surface.

RÉSUMÉ : Afin de développer une technologie d'amélioration du sol auto-cicatrisante, les caractéristiques de récupération de la résistance au cisaillement des argiles Kawasaki et Kaolin traitées au ciment ont été clarifiées au moyen d'une série de tests de cisaillement en boîte et d'une analyse par diffraction des rayons X sur poudre. Les principales conclusions sont les suivantes: 1) La récupération de la résistance au cisaillement dépend du type de sol du matériau de base, de la teneur en GGBS et de la pression verticale pour l'essai de cisaillement en caisson. L'argile Kawasaki traitée au ciment montre une forte récupération de la résistance au cisaillement, allant de 7,4% à 340,8%. 2) Quatre indices ont été proposés pour exprimer la récupération de la résistance au cisaillement de l'argile traitée au ciment. Le GGBS est efficace pour augmenter la résistance au cisaillement initiale, cependant, le GGBS a un effet négatif sur la récupération de la résistance de l'argile traitée au ciment. 3) La résistance au cisaillement de récupération consiste en un important composant de friction, bien que l'argile conventionnelle traitée au ciment ait un important composant de cohésion. On peut considérer que la réaction chimique du ciment sur la surface de cisaillement après cisaillement augmente la résistance fictive au lieu du composant cohésif. 4) On considère que la récupération de la résistance au cisaillement de l'argile traitée au ciment auto-cicatrisante est due à l'augmentation de l'Ettringite et de l'hydrate d'aluminate de calcium par l'hydratation du ciment non hydraté sur la surface de cisaillement.

KEYWORDS: cement mixing, unconfined compressive strength, strength recovery, X−ray diffraction.

1 INTRODUCTION

In Japan, the conventional ground solidification technique, which mixes an improvement material such as cement into soft ground, has been widely used to improve the bearing capacity of the ground and prevent from soil liquefaction. Since large-scale earthquakes have frequently caused damage to natural slope and soil structure such a river reeve in recent years, sustainable and resilient ground improvement technology is needed. For concrete material, the self-healing concrete using bacteria and restorative material has been developed. However, in the field of geotechnical engineering, there are few previous studies on the self-healing of geomaterial (e.g., DeJong et al., 2013 and Hata et al., 2018).

From these backgrounds, our research group investigated the self-heling characteristics of the shear strength for cement-treated clay subjected to cyclic loading during curing period by unconfined compression test (Takayama et al, 2019). In this paper, in order to develop a self-healing ground improvement technology, the recovery characteristics of the shear strength for cement-treated soil was clarified by means of a series of box shear tests which was subjected to an initial shear during curing period on cement-treated clay. In addition, in order to investigate the shear strength recovery in terms of the chemical components of soil and cement mixture, powder X-ray diffraction (XRD) was performed against chemical substances on the shear surface of cement-treated clay.

2 EXPERIMENT OUTLINE

2.1 *Sample preparation and experimental procedure*

Soft clays dredged at Kawasaki area in Japan (called "Kawasaki clay" hereafter) and Kaolin clay were used as the base material. Table 1 shows the physical property of Kawasaki clay and Kaolin clay. Figure 1 shows the grain size distribution of Kawasaki clay and Kaolin clay. Table 2 summarizes experimental conditions. Ordinary Portland cement (OPC) was mixed at 10 % of the dry weight of the base material. In addition to cement, Ground-granulated blast furnace slag (GGBS) powder was mixed as auxiliary material. GGBS has a latent hardening property to increase a long-term strength, therefore three GGBS contents of 0%, 5% and 10% of the dry weight of the base material was set to enhance the strength recovery. For the preparation of the test specimen for the diameter of 60 mm and the height of 20 mm "Preparation of stabilized soil specimens without compaction (JGS T821)" was used. The initial water content of both clays were set to 100% to reach the shear strength to 100 kPa.

In order to case damage for cement-treated specimen, an initial shearing was introduced at 7 days after preparing specimen, assuming that the strength development of the cement-treated clay was completed in 28 days. The shear displacement for the initial shearing was 3 mm. After the initial shearing, specimen was re-cured again in a humid room at a temperature of 20 °C. Finally, after 21 days for re-curing process, specimen was subjected to a secondary shear test to examine the effect of initial

Table 1. Physical property of Kawasaki clay and Kaolin clay.

	Kawasaki clay	Kaolin clay
Soil particle density [g/cm^3]	2.66	2.63
Liquid limit [%]	51.0	82.1
Plastic limit [%]	25.6	34.7
Plastic index	25.4	47.4
Ignition loss [%]	6.0	13.0

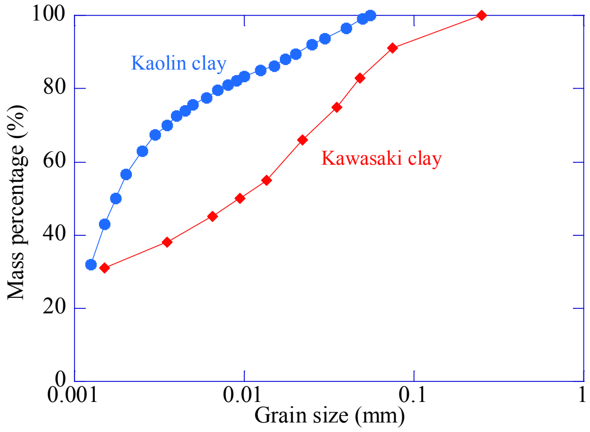


Figure 1. Grain size distribution of Kawasaki clay and Kaolin clay.

shearing on the strength recovery characteristics. The vertical pressure for constant pressure box shear test was 100 kPa for Kawasaki clay and 50, 100, and 150 kPa for Kaolin clay.

For comparison, a series of box shear tests were also performed on specimens without the initial shearing that cured for 7 days and 28 days respectively. Thereafter, the specimen with the initial shearing is referred to as "initial shear specimen", and the specimens without the initial shear are referred to as "7 days shear specimen" and "28 days shear specimen" respectively.

2.2 *Powder X-ray diffraction*

In order to investigate the relationship between the chemical composition of specimen and the strength recovery characteristics, we quantified chemical compounds on the shear surface of the cement-treated specimen after box shear testing by using the powder X-ray diffraction (XRD). Table 3 shows a list of chemical compounds set up as search targets for the XRD analysis. Kaolinite is the main component of Kaolin clay. From C3S to Periclase, shaded area by light gray in Table 3, are compounds expected to be contained in cement and GGBS while from calcium hydroxide to C-A-H, shaded area by dark gray in Table 3, are compounds that were formed the chemical reaction of cement and water during curing.

3 STRENGTH RECOVERY CHARACTERISTICS

3.1 *Test result of box shear test*

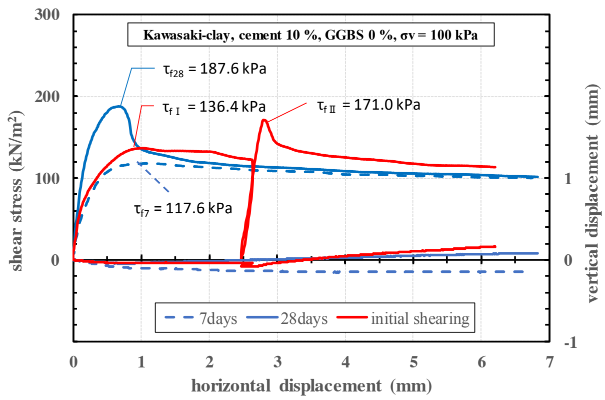
Figure 2(a) and 2(b) shows the relationship between shear stress, vertical and horizontal displacement for cement-treated Kawasaki clay and Kaolin clay with GGBS 0 % respectively. It is noted that blue lines indicated the test results of 7 and 28 days shear specimens without initial shearing, respectively. 28 days shear specimen shows clear peak strength due to additional curing compared with 7 days shear specimen. It can be seen that a clear peak shear strength was observed for initial and secondary shearing, and the peak shear strength for secondary shear exceeded residual strength for the initial shear. Especially for cement-treated Kawasaki clay, the peak shear strength for secondary shear exceeded peak strength for 7 days shear specimens and it is comparable to that for 28. For cement-treated Kaolin clay, however, it was smaller than peak strength for 7 and 28 days shear specimens for cement-treated Kaolin clay.

Table 2. Sample preparation and experiment condition.

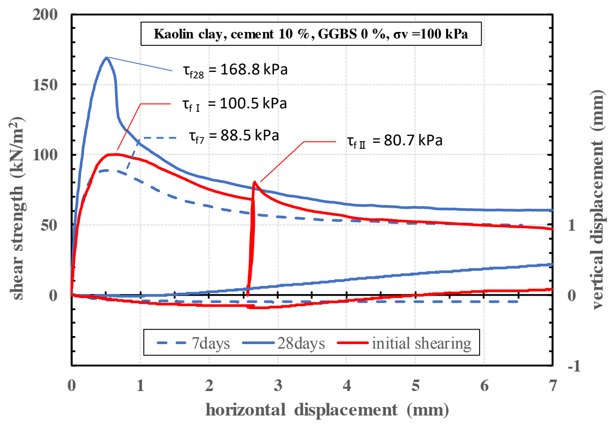
Base material	Kawasaki clay	Kaolin clay
Initial water content	100 %	
Cement	Ordinary Portland Cement	
Cement content	10 %	
Auxiliary material	Ground-granulated blast-furnace slag (GGBS) powder	
GGBS content	0, 5, 10 %	
Curing condition	a humid condition and 20 °C.	
The day for initial shearing	7 days	
Initial shear displacement	3 mm	
Vertical pressure	100 kPa	50, 100, 150 kPa

Table 3. Search chemical components of XRD.

name	formula	name	formula
Kaolinite	$Al_4Si_4O_{10}(OH)_8$	C3S	Ca3SiO5
C2S	Ca2SiO4	C3A	$Ca_3Al_2O_6$
C4AF	$Ca_4Al_2Fe_2O_{10}$	Calcium Oxide	CaO
Gypsum	$CaSO_4$-nH_2O	Periclase	MgO
Calcium hydroxide	$Ca(OH)_2$	Calcite	$CaCO_3$
Ettringite	$3CaO$-Al_2O_3-$3CaSo_4$-$32H_2O$	C-A-H	$3CaO$-Al_2O_3-$6H_2O$



(a) Kawasaki clay



(b) Kaolin clay

Figure 2. The relationship among shear stress, vertical and horizontal displacement.

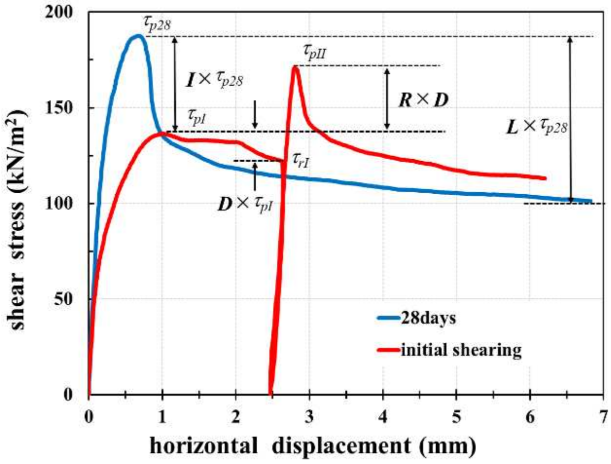


Figure 3. The definition to express shear strength recovery.

Therefore, it can be characterized that the strength recovery was confirmed for both cement-treated Kawasaki clay and Kaolin clay and the magnitude of strength recovery of cement-treated Kaolin clay is small compared to cement-treated Kawasaki clay. Takayama et al. (2019) also investigated the effect of cyclic loading at the early stage of curing period on the strength recovery of cement-treated Kaolin clay. They reported that the unconfined compressive strength increases with increasing GGBS content when cyclic loading is subjected to cement-treated Kaolin clay at the early stage of curing period (such as 7 curing day).

Vertical displacements of cement-treated Kawasaki clay and Kaolin clay during shearing is very small irrespective of curing day and with/without an initial shearing, which means small volume change in box shearing. It is suggested that undrain condition is kept in shearing for cement-treated clay.

3.2 *Recovery index*

In order to investigate the effect of initial shearing on the strength recovery in detail, the following indexes were introduced using initial shear peak strength τ_{pI}, initial shear residual strength τ_{rI}, secondary shear peak strength τ_{pII} and 28 days shear peak strength τ_{p28} as shown in Figure 3.

1) Increase rate I

$$I=\left(\frac{\tau_{p28}}{\tau_{pI}}-1\right)\times 100[\%] \quad (1)$$

The increase rate I indicates how much strength increased from 7 day to 28 day by curing without initial shearing. Large I means large strength increase due to secondary curing from 7 days to 28 days.

2) Decrease rate D

$$D=\left(1-\frac{\tau_{rI}}{\tau_{pI}}\right)\times 100[\%] \quad (2)$$

The decrease rate D indicates how much strength decreased at the end of the initial shearing from the peak shear strength. Large D means large strength decrease due to the initial shearing.

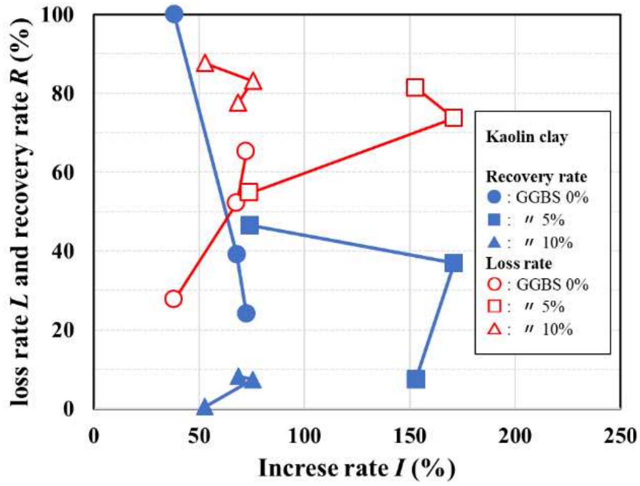
3) Loss rate L

$$L=\left(1-\frac{\tau_{rII}}{\tau_{p28}}\right)\times 100[\%] \quad (3)$$

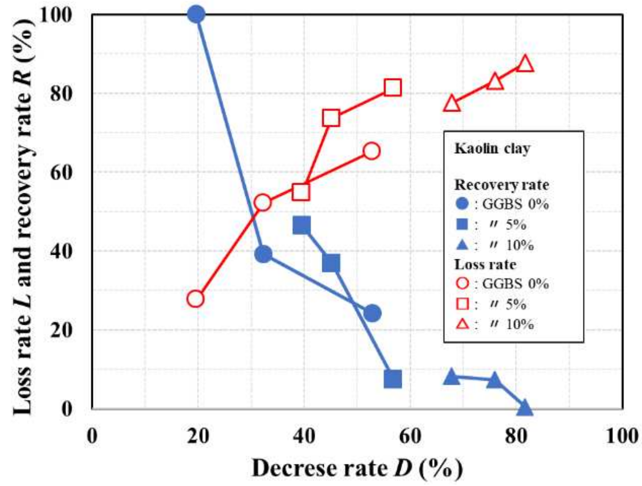
The loss rate L indicates how much shear strength decreased at the end of shearing, residual state. Large L means large strength decrease from peak state to residual state.

Table 4. Recovery index.

Specimens	GGBS (%)	Vertical pressure (kPa)	Increase Rate I (%)	Decrease Rate D (%)	Loss Rate L (%)	Recovery Rate R (%)
Kawasaki clay	0	100	37.5	10.5	8.9	340.8
	5	100	134.4	36.7	51.8	135.7
	10	100	88.8	56.6	69.5	25.1
Kaolin clay	0	50	72.4	52.9	65.3	24.2
		100	67.9	32.3	52.2	39.2
		150	38.2	19.6	27.6	100.0
	5	50	153.0	56.8	81.3	7.4
		100	171.0	45.2	73.6	36.9
		150	74.1	39.5	54.7	46.5
	10	50	52.7	81.6	87.7	0.5
		100	75.5	76.0	83.1	7.4
		150	68.5	67.8	77.6	8.2



(a) Loss rate L and recovery rate R against increase rate I



(b) Loss rate L and recovery rate R against decrease rate D

Figure 4. The relationship among recovery indexes for Kaolin clay.

4) Recovery rate R

$$R=\left(\frac{\tau_{pII}-\tau_{rI}}{\tau_{pI}-\tau_{rI}}\right)\times 100[\%] \quad (4)$$

The numerator of Equation (4) indicates the increase in shear

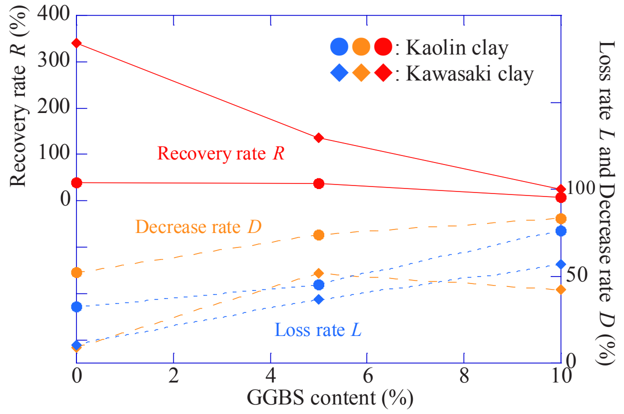


Figure 5. Mohr-Coulomb yield criterion of cement treated Kaolin clay with GGBS 0 %.

strength before and after re-curing, and the denominator of Equation (4) indicates the decrease in strength from the peak shear strength to ending state at initial shearing, which is equal to the decrease index *D*. Large *R* means large strength recovery compared with *D*.

Table 4 summarizes the result of four indexes for cement-treated Kawasaki and Kaolin clays. In order to examine the relationship between each index, Figure 4 shows diagrams among increase rate *I*, decrease rate *D*, loss rate *L* and recovery rate *R*. From Figure 4 (a), there is no strong correlation between *I* and *L* and *R*. However, from Figure 4 (b), as the decrease rate *D* increases, the loss rate *L* increases and the recovery rate *R* decreases. Therefore, the degree of strength recovery for cement-treated clay is determined by the residual strength at the end of initial shearing, which is one of factors to determine the decrease rate *D*. It can be characterized that strong strength recovery was achieved as the recovery rate from 7.4 % to 340.8 %, especially for Kawasaki clay. Moreover, from Figure 4(b), as GGBS content increases, the recovery rate *R* decreases and the loss rate *L* increases. This is because the peak shear strength for initial shear and the 28-day specimens increases significantly with increasing GGBS content, however GGBS has little effect on peak strength for secondary shear. Table 5 summarizes that the peak shear strength at the vertical pressure of 100 kPa for cement-treated Kaolin clay with different GGBS content. It can be considered that GGBS is effective for increasing the initial shear strength, however, GGBS has little effect on the strength recovery.

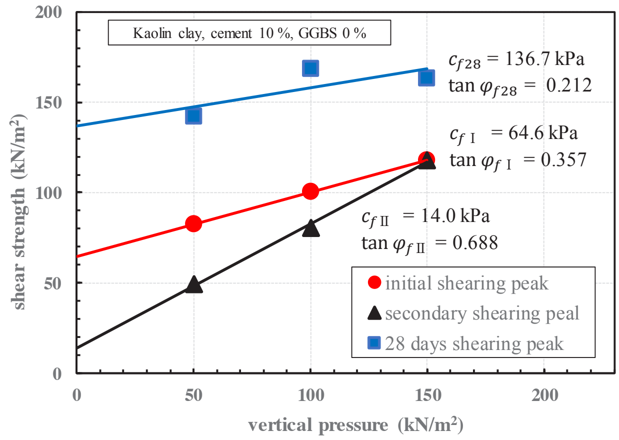
In order to compare strength recovery in terms of the soil type and GGBS content, the recovery rate *R*, loss rate *L* and decrease rate *D* of cement-treated Kawasaki and Kaolin clays are plotted against GGGBS content in Figure 5. The recovery rate *R* of cement-treated Kawasaki clay decreases sharply with increasing GGBS content while that of cement-treated Kaolin clay shows almost constant. Decrease rate *D* for both cement-treated Kawasaki and Kaolin clays increases with increasing GGBS content, however, the magnitude of *D* of Kaolin clay is 20% larger than that of Kawasaki clay. Loss rate *L* for both cement-treated Kawasaki and Kaolin clays increases with increasing GGBS content. It is characterized that recovery indexes depends on soil type and GGBS content, which suggests that strength recovery should be examined in terms of clay minerals of original material before cement mixing and chemical compounds due to hydration chemical reactions among clay minerals, cement and water.

3.3 *Mohr-Coulomb failure criterion*

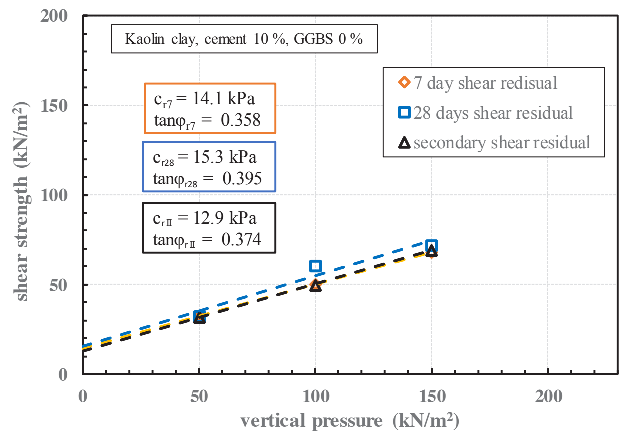
Figure 6(a) shows the Mohr-Coulomb failure criterion obtained from the box shear test results for cement-treated Kaolin clay with GGBS 0%. From Figure 6(a), without initial shear, the cohesion component at the peak state increased significantly from 7th day to 28th day. The *c* and ϕ for 28 days specimen are 136.7 kPa and 12.0 º respectively while those for 7 days specimen are 64.6 kPa and 19.6 º respectively. This is because the cohesion increases due to the cement chemical reaction during curing. However, when the initial shear was applied, the cohesion component reduced greatly while the internal friction angle increased greatly. The *c* and ϕ for initial shear specimen are 14.0 kPa and 34.5 º respectively. Therefore, it can be characterized that the cohesion component increases for the cement-treated specimen without an initial shear while the internal friction component increases for cement-treated specimen with an initial shear. It can be considered that the cement chemical reaction on shear surface after initial shear augments the fictional resistance instead of cohesive component.

Table 5. Shear peak strength of cement-treated Kaolin clay under vertical pressure 100 kPa.

		τ_{pI} (kPa)	τ_{p28} (kPa)	τ_{pII} (kPa)
GGBS (%)	0	100.5	168.8	80.7
	5	113.2	306.6	80.9
	10	304.8	534.9	90.3



(a) Peak state



(b) Residual state

Figure 6. Mohr-Coulomb yield criterion of cement treated Kaolin clay with GGBS 0 %.

Figure 6(b) shows the residual state at the horizontal displacement of 7 mm for cement-treated Kaolin clay with GGBS 0%. All test results range from 12.9 kPa to 15.3 kPa for cohesive component and from 16.7 º and 21.6 º for frictional angle. It can be seen that the residual state for cement-treated Kaolin clay is similar irrespective of initial shear and curing days.

4 XRD CHEMICAL COMPONENT ANALYSIS

Figures 7(a) and 6(b) show the mass ratio of chemical compounds obtained by XRD analysis for cement-treated Kawasaki and Kaolin clay. Those with a mass ratio of 1 % or less were collectively separated into “Others”.

For cement-treated Kawasaki clay, the mass ratio of Ettringite increase from 2.40 % to 3.57 % due to curing from 7 days to 28 days while Calcium Aluminate hydrate (C-A-H) also increases from 0.90 % to 3.02 %. This is due to the progress of cement chemical reaction during curing process. Moreover, for the initial shear specimen, the mass ratios of Ettringite and C-A-H are 4.15 % and 5.90 % meaning that the mass ratio of Ettringite and C-A-H increased in total 6.75 % compared to 7 days shear specimen. It is suggested that the strong shear strength recovery of cement-treated Kawasaki clay was confirmed by the chemical component analysis.

For Kaolin clay, the increase in the mass ratio of Ettringite and Calcite is not well observed after curing from 7 days to 28 days. However, for the initial shear specimen, the mass ratios of Calcite increases to 11.5 % compared to 7 and 28 days shear specimens. It is considered to be one of reasons why weak shear strength recovery was observed for cement-treated Kaolin clay.

5 CONCLUSIONS

In order to develop a self-healing ground improvement technology, the recovery characteristics of the shear strength for cement-treated Kawasaki and Kaolin clays was clarified by means of a series of box shear tests. In addition, in order to investigate the shear strength recovery in terms of chemical components of soil and cement, powder X-ray diffraction analysis was performed against chemical substances on the shear surface of cement-treated Kawasaki and Kaolin clays. The main conclusions are as follows:

1) The specimen, which has an initial shearing on 7 day and re-curing for 21 days, recovers its shear strength from the initial shear residual strength. The magnitude of shear strength recovery depends on the soil type of base material, GGBS content and the vertical pressure for box shear test. Cement-treated Kawasaki clay shows strong shear strength recovery ranging from 7.4 % to 340.8 %.

2) Four indexes such as an increase rate *I*, decrease rate *D*, loss rate *L* and recovery rate *R*, were proposed to express the shear strength recovery for cement-treated clay. Based on four indexes evaluation, the recovery rate *R* increases with decreasing loss rate *L* and decrease rate *D*. In addition, GGBS is effective for increasing the initial shear strength, however, GGBS has negative effect on the strength recovery of cement-treated clay.

3) In terms of the strength parameter, the shear strength recovery consists of a large friction component after an initial shearing although conventional cement-treated clay has a large cohesion component. It can be considered that the cement chemical reaction on shear surface after initial shearing augments the fictional resistance instead of cohesive component.

4) The shear strength recovery of self-healing cement-treated clay is considered to be due to the increase of Ettringite and Calcium Aluminate Hydrate by the hydration of Unhydrate cement on the shear surface.

6 ACKNOWLEDGEMENTS

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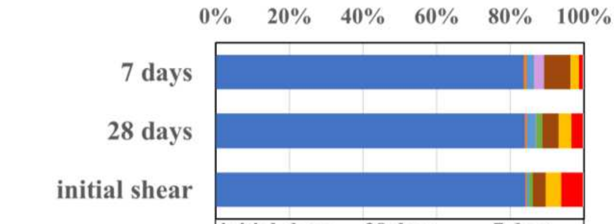
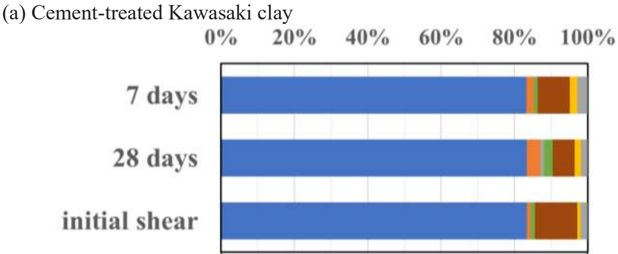


	initial shear	28 days	7 days
Mineral	83.9	83.9	83.9
C3S	0.45	0.66	0.95
C2S	0.55	2.31	1.80
C4AF	0.05	0.16	2.80
Calcium hydroxide	1.00	1.65	0.10
Calcite	3.45	4.40	7.10
Ettringite	4.15	3.57	2.40
C-A-H	5.90	3.02	0.90
Others	0.30	0.33	0.39

(a) Cement-treated Kawasaki clay



	initial shear	28 days	7 days
kaolinite	83.3	83.3	83.3
C3S	1.01	3.61	2.04
C3A	0.05	1.00	0
Calcium hydroxide	1.23	2.41	1.04
Calcite	11.5	5.87	8.87
Ettringite	0.90	1.81	1.98
Others	1.93	1.82	2.87

(b) Cement-treated Kaolin clay

Figure 7. Mass ratio of chemical compounds by XRD.

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