

## Technical Paper

# A study on effect of silica fume and surface penetrant on bonding strength for overlaying

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(Received May 18, 2018; Revised March 10, 2019; Accepted April 16, 2019; Published online June 30, 2019)

**Abstract:** Currently, concrete structures are at risk of deteriorating conditions due to their exposure to various severe environments. Repairing and strengthening these structures is a better solution than replacing them by new constructions. As a retrofitting method, the top/bottom surface overlaying or jacketing method is commonly used around the world. In this method, polymer cement mortar (PCM) is sprayed/troweled onto the surfaces of concrete, and PCM adhesive strengthens the concrete structures. However, the concrete-PCM interface is considered to be the weakest part of a structure, and it may cause brittle fracture. This study focused on how this interface can be strengthened more effectively to prevent brittle fractures and investigated the effects of applying surface penetrant and silica fume from the perspective of chemical reactions. In addition, the reaction time relationship between PCM hydration and surface penetrant application time is also investigated based on a preliminary experiment. Mixing silica fume into PCM was conclusively found to strengthen the interface bonding strength in some conditions. Applying surface penetrant to an interface could decrease the interface strength if the application time was inappropriate. However, the results of an application time experiment indicated that it is possible to strengthen the interface via the surface penetrant.

**Keywords:** Overlaying method, interface strength, chemical bonding, silica fume, surface penetrant, PCM.

## 1. Introduction

During the period of high economic growth after World War II in Japan, numerous large construction projects evolved, and structures were constructed with concrete. These concrete structures incurred damage from exposure to severe environmental conditions with various loading impacts, which reduced their lifespan. Maintenance or rehabilitation is the most suitable solution, both economically and environmentally, for the restoration of deteriorated concrete structures. Therefore, the need to repair, strengthen, and adapt structures to new loads is increasing [1]. As a retrofitting method, the top/bottom surface overlaying method (or the jacketing method) is commonly employed around the world [2]. In this

method, cementitious material, such as polymer cement mortar (PCM), is sprayed or troweled onto the surface of concrete structures to increase the cross-sectional area of structures and improve the construction durability. This method is dependent on the adhesive force of PCM. Among all cementitious materials, polymer modified cement mortar provides excellent adhesion at the interface because polymer films surround the hydration products and aggregates. Coalescence of polymer particles fills all pores, reduces the porosity, and increases the adhesive strength [3,4].

However, the shear stress at the interface between the substrate concrete and the repairing material, which is induced by the forces carried by the repairing material, causes debonding fracture at the interface [5]. One approach to prevent brittle debonding fracture is to increase the interface strength; understanding the process of increasing the interface strength may enable the development of a new retrofitting method. This study focused on how the interface can be strengthened more effectively. Concrete and PCM are connected at the interface by relying on the micro filler and anchoring effect of PCM. The

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smaller particles of PCM fill the rough concrete surface gaps, which is referred to as the micro filler effect; these particles harden due to hydration and resist stresses, which is referred to as the anchoring effect. Chemical bonding does not occur across the interface despite the chemical bond between the concrete and the PCM. Therefore, the formation of chemical bonds at the interface is considered to strengthen the interface bonding strength. This study focused on generating interface chemical bonding by applying two types of materials: silica fume and surface penetrant.

Silica fume is also known as micro silica, which is an ultrafine powder collected as a byproduct of silicon and ferrosilicon alloy production. Silica fume provides greater cohesiveness, less segregation, and reduced bleeding in concrete and consists of microspheres with a mean diameter of approximately 0.15 microns and a very specific surface area (15,000-25,000 m<sup>2</sup>/kg) [6]. The size of these microspheres is approximately 100 times smaller than the average size of cement particles. Due to its extreme fineness, silica fume is expected to strengthen the interface by the micro filler effect [7]. The high silica content in silica fume can produce C-S-H crystal by combining with calcium hydroxide in the water supplied condition [8], which can enhance the interface bonding strength. As a supplementary material, surface penetrant was employed. To form chemical bonds between the existing concrete and the coating of PCM, old concrete and PCM hydration must be considered. Chemical bonds are created simultaneously causing reactions in both materials. However, the substrate concrete cannot easily undergo chemical reactions. Thus, we focused on the use of a surface penetrant material. This material is usually employed to treat a

cured concrete surface as a surface modifier to protect the concrete surface. In this study, however, the surface penetrant was employed to improve the interface instead of protecting the surface. The formation of a chemical bond between the existing concrete and the PCM coating is expected by applying a surface penetrant to the interface.

The objective of this study is to confirm the effectiveness of applying a surface penetrant and silica fume to enhance the tensile strength and shear strength at the interface between concrete and PCM. The environmental conditions, such as temperature and moisture, will affect the strength of the interface between concrete and PCM. However, the effects of the environmental conditions are not considered in this study.

## 2. Outline of Test

### 2.1 Test method

To measure the tensile strength, a splitting tensile test was conducted using a cubic specimen according to the process described by Li or Rashid [9,10], as shown in Fig. 1(a). The splitting tensile strength can be evaluated based on elastic theory. To determine the interface shear strength, a single-surface shear test was performed in this study. The jig used in the test is shown in Fig. 1(b), and the load was applied on the jig using a compressive machine. The compressive strength of concrete and PCM were measured using cylindrical specimens with dimensions of 100x200 mm for concrete and 50x100 mm for PCM based on JIS A 1108 and JSCE-F506, respectively [11,12].

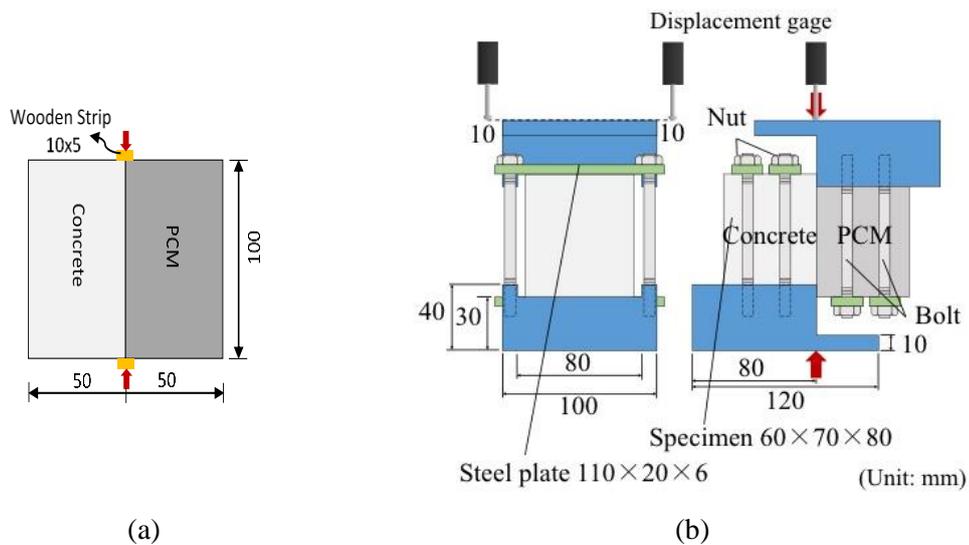


Fig. 1 – Schematic of (a) splitting tensile test and (b) direct single-surface shear test

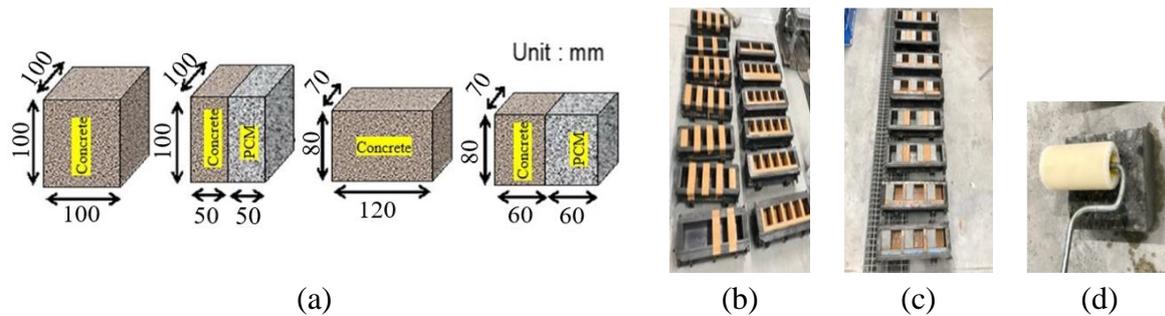


Fig. 2 – Preparation of the specimens in the experiment: (a) specimen sizes, (b) molds for the substrate concrete, (c) molds for overlaid PCM, and (d) application of the surface penetrant

Table 1 – Number of specimens and types of tests performed

Specimen types		Test types		
		Compressive	Splitting tensile	Shear
Monolithic specimens	Concrete (A)	3	3	3
	PCM (B)	3	3	3
	Silica fume PCM (2.5%) (C)	3	3	3
	Silica fume PCM (5%) (D)	3	3	3
Composite specimens	(A+B)	--	3	3
	(A+C)	--	3	3
	(A+D)	--	3	3
	(A+B) with surface penetrant	--	3	3
	(A+C) with surface penetrant	--	3	3
	(A+D) with surface penetrant	--	3	3

Table 2 – Mix proportion of concrete

Maximum aggregate (mm)	Slump (cm)	W/C (%)	s/a (%)	Amount (kg/m <sup>3</sup> )				
				Water	Cement	Sand	Aggregate	AE agent
25	9.0	43.0	38.0	164.3	382.1	683.8	1128.0	0.0382

Table 3 – Mix proportion of PCM

Type	Water/PCM (%)	Silica fume (% of PCM mass)	Superplasticizer (% of PCM mass)
Normal PCM	14.0	0	0
PCM (2.5% of silica fume)	14.0	2.5	0.5
PCM (5% of silica fume)	14.0	5.0	1.0

## 2.2 Specimen preparation

Schematics of the splitting tensile test specimen and shear test specimen are shown in Fig. 2(a); the dimensions of the cube specimen and the prism specimen are 100×100×100 mm and 120×70×80 mm, respectively. In the case of composite specimens, half size concrete specimens were constructed. Before casting the PCM, the concrete surface was roughened by sandblasting after 14 days of curing. PCM was overlaid onto the last half. Each mold for the substrate concrete and for overlaying the PCM are shown in Fig. 2(b) and 2(c), respectively. Silica fume was mixed into the premixed PCM right before mixing with water. Surface penetrant was applied right

before overlaying and then returned to the molds, as shown in Fig. 2(d). The number of specimens that were tested in this study is shown in Table 1.

High-early-strength Portland cement, fine/coarse aggregate with a maximum aggregate size of 25 mm and AE agent were utilized for the substrate concrete. Premixed PCM that contains poly acrylic ester (PAE), reactive type sodium silicate surface penetrant and silica fume supplied by the company were employed as a repair material. The water/binder ratio of the PCM was previously determined to be 14% in advance. We controlled the silica fume amount as the parameter in the mix. The mix

proportion of the concrete and the PCM are shown in Table 2 and Table 3, respectively.

### 2.3 Mixing silica fume

Silica fume supplied by the Denka Company was utilized in this study. In general, silica fume is used as a supplementary cementitious material to increase the strength and durability. The use of substantial amount of silica fume causes a lack of water because it consumes a substantial amount of water due to its small particle size. A pozzolanic reaction also consumes water. Therefore, large amount of silica fume may cause material stagnation, whereas a very small amount of silica fume hinders the formation of the required C-S-H crystals at the interface, which does not satisfy the interface strengthening requirement [13]. In this study, silica fume of 2.5% and 5% of the PCM mass were mixed with pre-mixed PCM that contains PAE to prepare silica fume PCM. Silica fume was mixed into PCM powder right before mixing with water, and the mixed materials were hydrated. The W/B ratio of 14% was employed for the mix. Superplasticizer of 0.5% and 1.0% of the PCM mass were mixed into the 2.5% silica case and the 5% silica case, respectively, to prevent the formation of silica fume lumps.

### 2.4 Applying surface penetrant

In this study, reactive type (silicate based) surface penetrant was utilized in the test. The main component of the surface penetrates was “sodium silicate”. For each experimental condition, two cases were considered: with surface penetrant and without surface penetrant. Surface penetrant was applied

right before overlaying PCM; when it was applied to the roughed concrete surface, the surface was wetted according to Guideline on Design and Application Methods of Silicate-based Surface Penetrants used for Concrete Structures [14]. We sprayed water on the roughened substrate concrete surface and applied surface penetrant after water stagnation had disappeared.

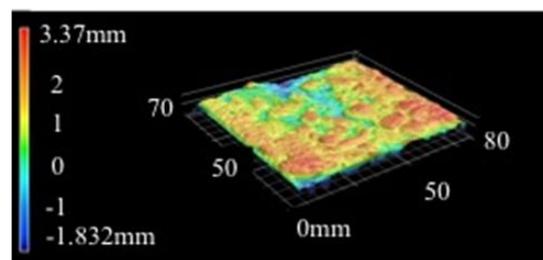
### 2.5 Surface roughness

Generally, a rough surface has a larger surface area for PCM attachment than a smooth surface, and the roughness level highly influences the interfacial strengths. Water jetting and sand-blasting techniques are considered to be the most suitable techniques for roughening the substrate concrete [15]. In this study, sandblasting techniques are applied to roughen the substrate concrete surface. Fig. 3(a) shows the measured apparatus, and Fig. 3(b) shows an example of the measured surface roughness.

The sampling length, namely cut-off level is used to separate roughness and waviness. Cut-off length is defined as 0.08 mm, 0.25 mm, 2.50 mm, and 8.0 mm for surface of metal, whereas for concrete surface 80 mm cut-off length is proposed by past studies [16] which is also used in our study. The average measured arithmetic roughness ( $R_a$ ) was 0.48 mm and ranged from 0.37 to 0.55 mm. In previous investigations, a sand blasting method for treating substrate concrete surface was adopted and the value of  $R_a$  was 0.37 mm [17,18]. In this study, the surface roughness is considered to have the same level of influence on the interface; we did not focus on the surface roughness.



(a)



(b)

Fig. 3 – Measurement of the surface roughness: (a) measured apparatus, (b) measured surface

## 3. Test Results

### 3.1 Fracture mode

The pure interface fracture mode (I) and interface composite fracture mode (I-P) of the composite specimens are shown in Fig. 4(a) and 4(b), respectively. The yellow part denotes the attached concrete, and the other part is fractured at the interface. According to the position of fracture on the surface of the specimens, six types of fracture modes are named, of which the concrete part fracture (C) and

PCM part fracture (P) are cohesion fractures that occur either in the concrete or the PCM part, while the interface fracture (I) is the adhesion fracture that occurs at the interface. The composite fracture modes include: mixed concrete and interface (C-I), in which some amount of substrate concrete attached on the PCM side, mixed interface and PCM (I-P), where some amount of PCM attached to the substrate concrete and mixed concrete and PCM (C-P), where the fracture occurs in both the concrete and the PCM layer. The mixed fracture (C-P), where no interfacial

fracture occurred, is the most desirable fracture mode, because this study intends to increase the interfacial bond strength. However, all composite specimens with surface penetrant applied exhibited interface fracture (I). The fracture modes of specimens without surface penetrant are shown in Table 4. The tensile test results indicate that the fracture mode of normal PCM includes interface fractures: pure interface fracture (I) and interface-PCM composite fracture (I-P). However, the specimen with 2.5% silica exhibits only one interface fracture of the interface-PCM composite (I-P) and does not include any pure interface fractures (I). The specimen with 5% silica does not include any interface fractures. Thus, the fracture mode shifted from the interface (I), such as (I) and (I-P), to the one with concrete (C) such as (C) and (C-P), with increased mixing of silica fume. For the shear test, two cases of interface fracture, named (I) and (I-P), were observed in specimens with normal PCM. Similarly, 2.5% silica also represents two cases of interface fracture, named (C-I) and (I), while all 5% silica specimens showed frac-

ture in concrete only, such as (C) and (C-P). In addition, normal PCM has only one case of concrete part fracture (C-P), and the specimen with 2.5% silica has two cases of concrete part fractures, such as (C-I) and (C-P). Therefore, the fracture modes shifted from the interface to the concrete-PCM composite fracture mode in the shear test. The fracture mode differed among the specimens with the same condition. This difference is considered to have occurred because concrete is a composite material. In particular, the positions of the interface aggregates significantly affect the interface strength. In addition, the test method may have caused the difference in the fracture mode. By mixing silica fume with PCM, the fracture mode shifts from the interface to the concrete part. This result of the specimens with surface penetrant reveals that surface penetrant weakens the interface bonding strength and causes propagation of the interface crack between concrete and PCM. Applying the surface penetrant right before performing the overlaying process does not strengthen the interface bonding.

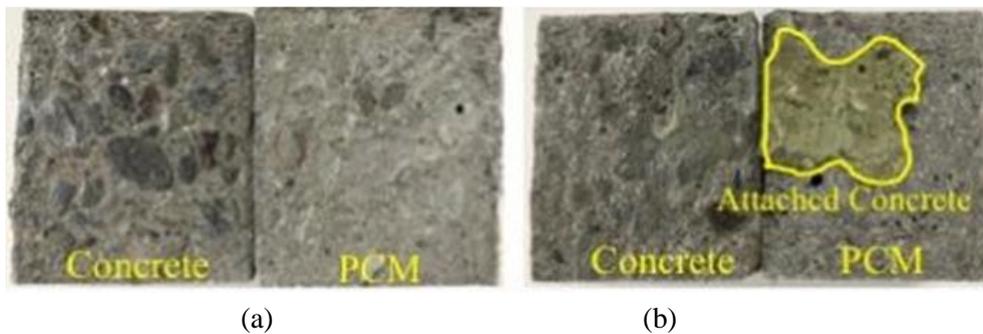


Fig. 4 – Fracture modes of the specimen: (a) interfacial fracture (I), (b) composite fracture (I-P)

Table 4 – Fracture modes of composite specimen without surface penetrant

Composite material	Fracture mode	
	Tensile test	Shear test
PCM1	C-P	C-P
PCM2	I	I
PCM3	I-P	I-P
2.5% silica 1	I-P	C-I
2.5% silica 2	C-P	I
2.5% silica 3	C-P	C-P
5% silica 1	C-P	C-P
5% silica 2	C	C-P
5% silica 3	C-P	C-P

### 3.2 Fracture energy

Fig. 5 shows the relationship between the shear stress and displacement. The specimens with surface penetrant applied showed similar slopes, as shown by the dotted lines in the figure. This similarity may correspond to the specimens being fractured in the

same fracture mode as the interface fracture (I). Regarding the composite specimens without surface penetrant, the curves show an entirely different tendency, probably because these fracture modes differed. Some specimens fractured at the interface, and other specimens fractured in composite fracture mode. Thus, some fracture modes are affected by the concrete strength, and other fracture modes are affected by the PCM strength. The fracture energy is obtained from the area under the stress-displacement curve (Fig. 5), considering the ascending part to the peak stress level. The calculated fracture energy is shown in Table 5. The table reveals that the interface fracture, as denoted by I in the table, corresponds to a lower fracture energy than that of other fracture types. The concrete PCM composite fracture mode corresponds to a fracture energy that is higher than that of the other fracture modes, as identified by (C-P) in the table. Thus, the fracture energy is possibly affected by the fracture mode. The fracture energy of specimens with silica fume is more than that without silica fume. The result of “2.5% silica 1” indicates

that the fracture energy increases as the interface fracture shifts to the concrete or the PCM part because its fracture mode is concrete-interface composite fracture (C-I), and the fracture energy became higher than that of the other pure interface fractures

(I) and smaller than that of the other concrete-PCM composite fractures (C-P). Note that if the fracture occurs in a couple of surfaces, then the fracture energy condition may become more complex and difficult to estimate, as in the result of “5% silica 1”.

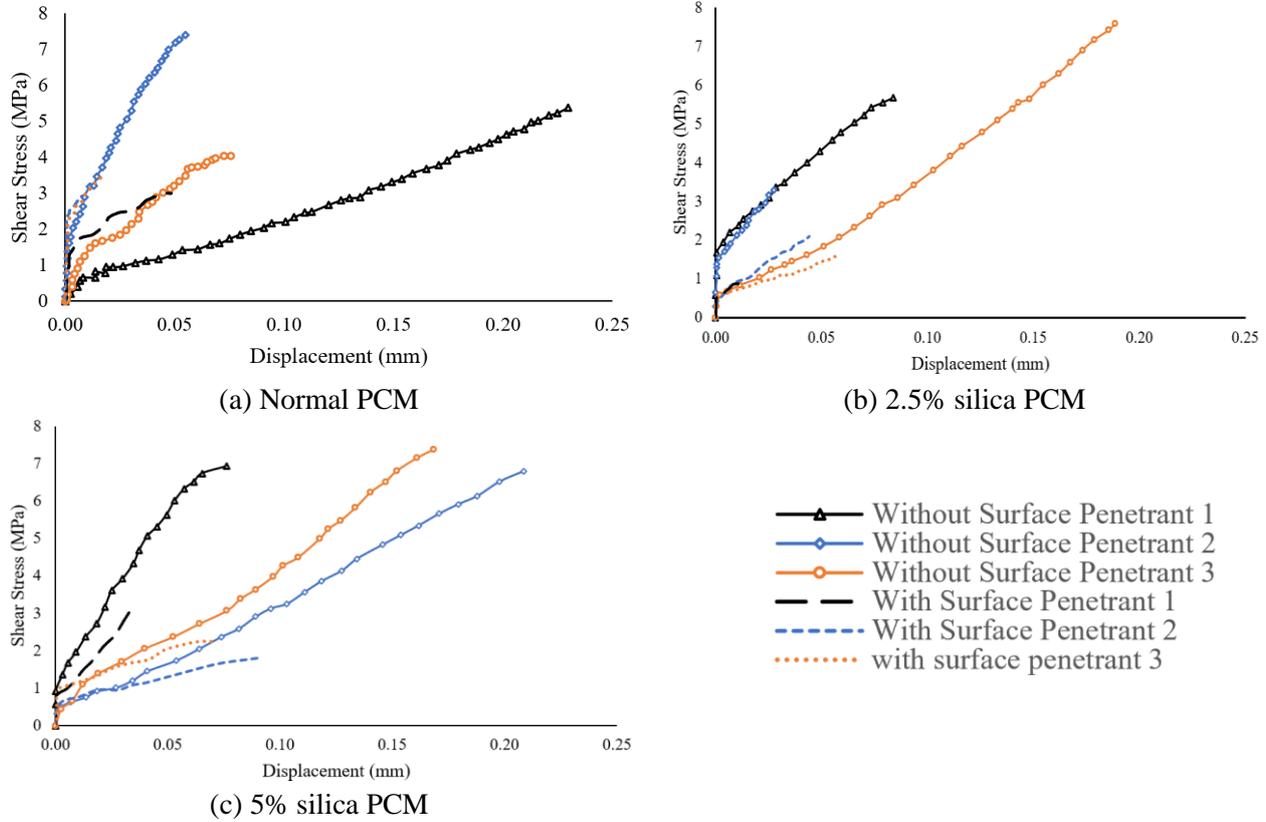


Fig. 5 – Relationship between shear stress and displacement on composite specimens

Table 5 – Fracture energy

Composite material	Fracture mode of shear test	Shear fracture energy (N/m)
PCM1	C-P	642.10
PCM2	I	242.50
PCM3	I-P	186.45
2.5% silica 1	C-I	323.21
2.5% silica 2	I	63.78
2.5% silica 3	C-P	690.62
5% silica 1	C-P*	345.50
5% silica 2	C-P	733.26
5% silica 3	C-P	638.58

Note: \* indicated fracture in couple of surfaces.

### 3.3 Maximum stress capacity

All calculated maximum stress capacities of the compressive, tensile, and shear test results are shown in Table 6. The values in this table correspond to the average of the three specimens. The concrete compressive strength was similar to that of PCM. This comparable strength may have caused more interface or fracture to the PCM, because the concrete did not

fracture as usual and may have changed the stress conditions. Concrete was cured more than PCM because the overlaying curing was performed after the concrete curing. Therefore, concrete seems to become stronger. Even if the concrete strength was comparable to that of PCM, mixing silica fume shifted the fracture mode from the interface to the concrete part, as previously explained. If both the

substrate concrete and the overlaying material are stronger, then more interface fracture occurs because the interface becomes comparatively weaker. The strengthened interface by silica fume withstood higher pressure. As a result, if the substrate concrete is weaker than that of this study, such as actual damaged concrete structures, then less interface fracture may be observed. Generally, the fracture mode appears to affect the maximum strengths. In this study, the fracture mode and the maximum stress did not exhibit a distinct tendency. However, as small tendencies, we can comparatively find that concrete-PCM composite fractures showed higher stress capacities.

**3.4 Silica fume in monolithic PCM specimens**

Fig. 6 shows the influence of mixing silica fume for monolithic PCM specimens. The vertical axis corresponds to each maximum stress capacity, and the horizontal axis corresponds to the mixing ratio of silica fume. Fig. 6(a) shows the compressive test results, Fig. 6(b) shows the tensile test results, and Fig. 6(c) shows the shear test results. Regarding the compressive and shear tests, mixing 2.5% silica fume of PCM mass produced an increase in the compressive and shear strengths, although the tensile strength was

not increased. Alternatively, specimens mixed with 5% silica fume had nearly the same result as that of the original PCM or had a decreased in tensile strength. Silica fume particles are filled into the gaps of cement particles; they can be expected to strengthen the interface or the repairing material. As explained in Section 2.3, too much silica fume causes a lack of water, which causes material stagnation and may prevent cement hydration. Silica fume has less tensile strength than cement hydration material. A previous study [19] reported that split tensile strength decreases with an increase in the percentages of silica fume in concrete. Vikassrivastava [20] stated that the modulus of elasticity and the tensile strength of silica fume concrete resembles the referal concrete. As a result, the addition of 5% silica fume is considered to have decreased strength, especially tensile strength. We can conclusively state that the effect of silica fume on the strength of monolithic specimen is marginal. The results that show some difference in strength among different silica fume contents can be considered as experimental scatter. Regardless, continuous and quantitative research studies should be performed.

Table 6 – Compressive, tensile, and shear test results

Test type	Specimen type									
	A	B	C	D	Without surface penetrant			With surface penetrant		
					A+B	A+C	A+D	A+B	A+C	A+D
Compressive test (MPa)	40.17	39.81	44.23	39.51	--	--	--	--	--	--
Splitting tensile test (MPa)	3.32 (0.32)	4.44 (1.27)	4.31 (1.39)	3.14 (0.44)	2.96 (0.60)	3.42 (1.74)	3.83 (0.57)	2.53 (0.45)	2.88 (0.26)	2.77 (0.52)
Shear test (MPa)	5.28 (0.34)	7.31 (0.03)	8.34 (0.05)	7.34 (0.15)	5.57 (0.35)	5.49 (0.70)	7.03 (0.13)	2.98 (0.15)	1.43 (0.29)	2.39 (0.12)

Note: The meaning of A, B, C, and D are shown in Table 1. The parentheses' value corresponds to standard deviation.

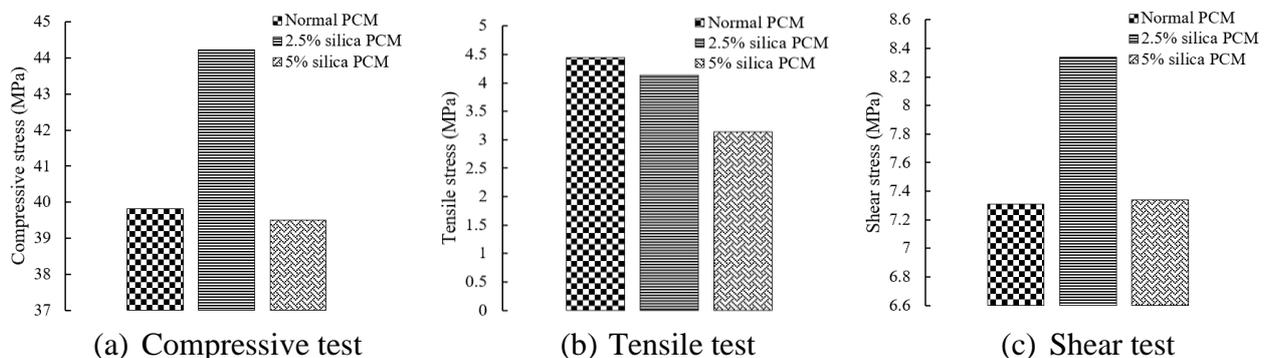


Fig. 6 – Silica influence in a monolithic specimen

### 3.5 Silica fume in composite specimens

Fig. 7(a) and 7(b) show the influence of silica fume on the tensile and shear strengths in composite specimens, respectively. The interface bonding strengths gradually increase with the addition of more silica fume, which reveals that the effect of silica fume on the interface strength is appreciable. This enhancement of the interface strength indicates that silica fume fills small voids at the interfacial zone, including surface layers of concrete and PCM sides. The tensile and shear strengths of the composite specimens are significantly affected by fracture surfaces. As explained in Section 3.1, the fracture mode of the composite specimens shifted from the interface to the concrete part with an increased mixing of silica fume. Thus, 5% silica appears to be the best case because this study intended to increase the interface bonding strength. However, if the fracture surface includes a concrete part or a PCM part, then the strength is also affected by the concrete strength or the PCM strength. As shown in Fig. 7, 2.5% silica strength is sometimes very similar to the concrete tensile strength and shear strength of 3.32 MPa and 5.28 MPa, respectively, and include a concrete part fracture. Note that if both the PCM and the interface

bonding strength are strong, then the maximum composite strength must be the concrete strength. In addition, even at an interface, the stress condition differed because the stiffness of the overlying material differed, and a stiffer overlying material causes higher stress concentration at the interface. On the other hand, the 2.5% silica case may be the stiffest, and the stress at the interface part is higher than that of the 5% silica case or the no silica case; as a result, interface fracture may have occurred despite the stronger interface.

### 3.6 Influence of the surface penetrant

Fig. 8(a) and 8(b) show the influence of the surface penetrant on the shear and tensile strengths, respectively. The figure shows that applying surface penetrant at the interface decreased the interface bonding strength. The decreasing ratio of the shear test was higher than that of the tensile test. This result may indicate that the shear strength is generally stronger than the tensile strength. We can conclusively state that applying surface penetrant to the interface right before overlaying PCM causes an interface fracture and decreases the interface bonding strength.

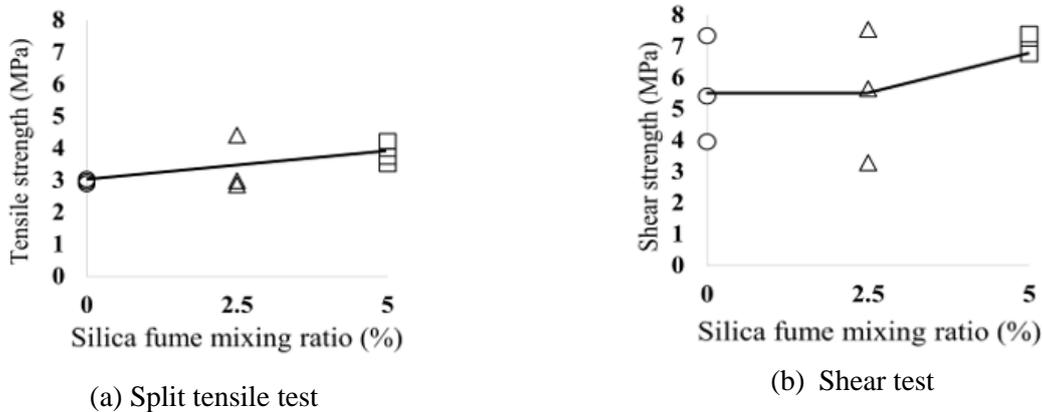


Fig. 7 – Influence of silica fume for the composite specimens

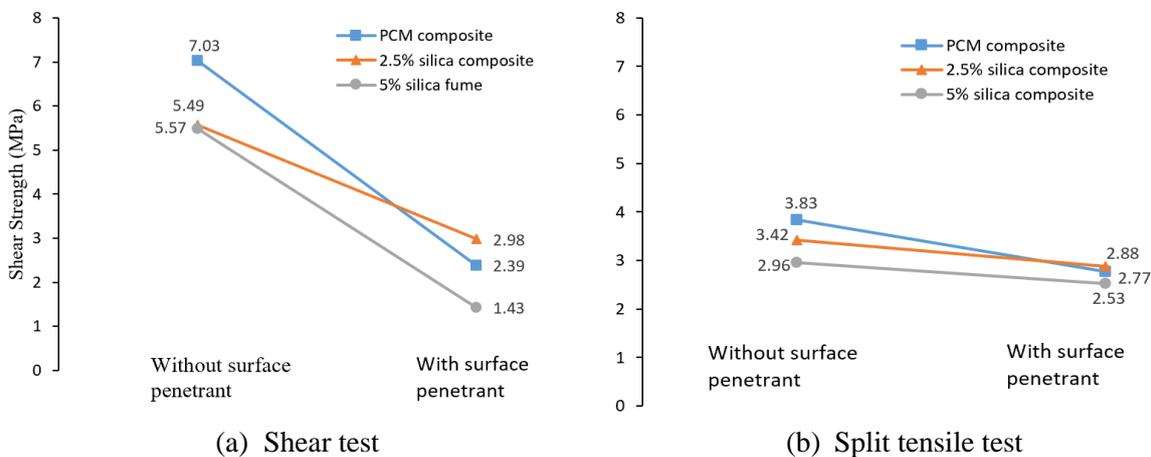


Fig. 8 – Influence of surface penetrant for the composite specimen

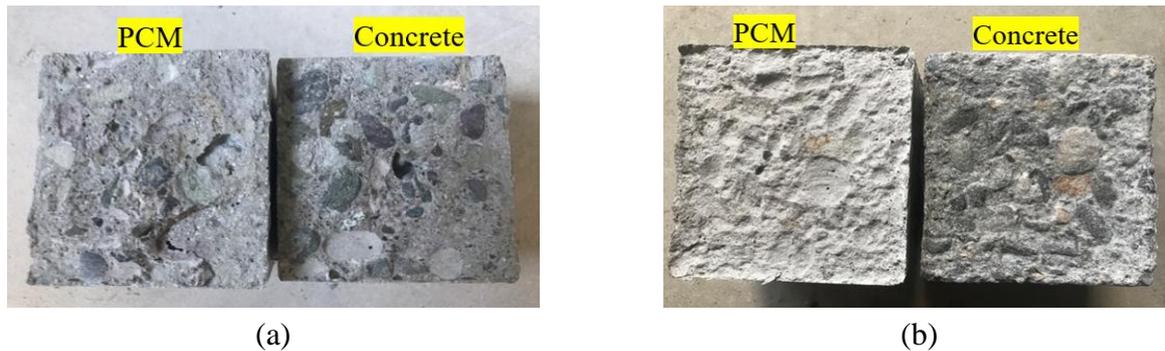
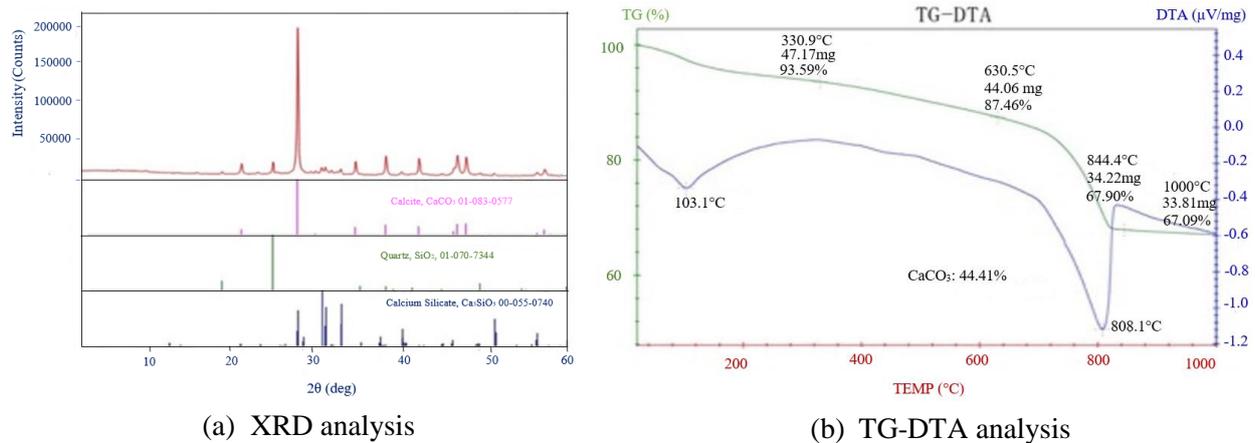


Fig. 9 – White compound at the interface of the composite specimen: (a) normal concrete-PCM composite, (b) concrete-PCM composite with surface penetrant



(a) XRD analysis

(b) TG-DTA analysis

Fig. 10 – Results of X-rays analysis of powdery white compound

### 3.7 Interface white compound

After fracturing, a white compound was observed that came from the interface of the specimens on which surface-penetrant was applied. Fig. 9(a) shows an ordinary PCM composite, and Fig. 9(b) shows a white compound on a PCM composite specimen in which the surface penetrant was applied. This compound is considered to prevent PCM adhesion and decrease the interface bonding strength. Before obtaining the decreased strength result, the generated compound was considered to fill the micro gaps inside concrete to produce the micro filler effect. Based on the results, however, we know that the presence of microcrystals sometimes decreases the interface bonding strength. According to Fig. 9(b), the compound is not located on the concrete surface but is located on the PCM surface because the PCM surface has a whiter appearance. In addition, the compound was also generated on the aggregate part. Therefore, the white compound appears to be the result of combining the surface penetrant with PCM.

X-ray diffraction (XRD) analysis of the white compound (in the form of powder) collected from the surfaces of the specimens with surface penetrant by polishing was performed. Fig. 10(a) shows the results of the XRD analysis of the powdery white compound, which confirm the presence of a large amount

of calcium carbonate ( $\text{CaCO}_3$ ) and a slight amount of silica sand ( $\text{SiO}_2$ ) and calcium silicate ( $\text{Ca}_3\text{SiO}_5$ ). Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA), as shown in Fig. 10(b), quantitatively measures the percentages of calcium carbonate ( $\text{CaCO}_3$ ) as 44%. Because the amounts of silica sand and calcium silicate are not large, we focused on calcium carbonate. Calcium carbonate is usually produced from the oxidation of calcium hydroxide. However, calcium hydroxide is usually generated in concrete at a concentration of less than 20%. Thus, 44% is considered a large concentration. Chemical reaction of concrete and PCM in presence of surface penetrant may promote taking  $\text{CO}_2$  in the air to make  $\text{CaCO}_3$  on the substrate concrete surface. Thus, in case of specimen with surface penetrant, higher percentages of  $\text{CaCO}_3$  can be found compared to the specimens without surface penetrant. Based on this discussion, it can be said that the white compound is calcium carbonate generated by the chemical reaction of fresh PCM with the surface penetrant.

## 4. Reaction Time Experiment

### 4.1 Objective

To realize chemical bonding at the interface, understanding and adjusting the chemical reaction time

between the PCM and the surface penetrant is important. A time lag exists between applying the surface penetrant and overlaying the PCM. If the overlaying is performed too early, then the chemical reaction of the surface penetrant may not proceed as rapidly as the hydration of the PCM. If overlaying is performed too late, then an excessively smooth surface may be produced by the surface penetrant that fills the surface gaps and prevents the anchoring effect of PCM. Therefore, timing of the PCM overlaying is one of the most important factors to gain a benefit from chemical bonds. When we focus on the PCM hydration time, the cement material inside the PCM intensively hydrates in two to four hours after mixing with water. As previously mentioned, the existing concrete re-reacts with the surface penetrant. However, the chemical reaction time between the concrete and the surface penetrant has not been intensively explored. The reaction may depend on the type of surface penetrant. Therefore, we need to know the exact surface penetrant reaction time to realize effective interface bonding. As a result, we conducted a preliminary experiment to evaluate the reaction time of the surface penetrant and reveal the mechanism by which the surface penetrant decreased the interface bonding strength.

## 4.2 Outline of test

### 4.2.1 Test method and specimen's preparation

This preliminary experiment was conducted by setting the overlaying time lag after the application of the surface penetrant as a parameter. To evaluate the best overlaying time, the interface bond strengths by the splitting tensile and the shear test as in Section 2.1 were compared. The dimensions of a final specimen for the split tensile test (cubic) and for the shear test (prism) are shown in Fig. 11(a-b) respectively.

In this experiment, only composite specimens were prepared. High-early-strength concrete was cast in advance, and after 14 days of curing the concrete, PCM was overlaid onto it. The mix design was the same as that of the experiment described in Section 2.2. The prism specimen dimensions were 120×70×80 mm in Section 2.2, whereas this preliminary test employed a specimen with dimensions of 100×70×80 mm. The concrete part with dimensions of 50×70×80 mm was placed at the bottom of the formwork, as shown in Fig. 11(c), and then PCM was placed on the concrete part. In this way, the quality of PCM placement can be better than the case of the main experiment shown in Section 2.2; in that experiment the PCM was placed on the vertical concrete surface, as shown in Fig. 2(c), but the former way is easier. In Section 2.2, substrate concrete was prepared by casting concrete in small molds. However,

this preliminary experiment prepared substrate concrete by cutting concrete prisms with dimensions of 100×100×400 mm. A more uniform interface can be expected because the cut surface had almost the same bleeding or aggregate conditions. Regarding the overlaying surface, a highly roughened surface is not always necessary, and a simple uniform surface is adequate for observing the pure reaction time of surface penetrant. By cutting the concrete to the half-size of the composite specimen, we prepared a uniform surface. The dimensions of the cut specimens are 100×100×50 mm and 70×80×50 mm. However, the concrete cutter uses water; this may promote calcium hydroxide (CH) eluviation along the concrete surface, which causes incomplete reaction and lower adhesion at the interface because the applied materials depend on the amount of CH. Therefore, the cut surfaces were grinded an additional 5 mm from the surface.

### 4.2.2 Time parameter

After 14 days of curing, surface penetrant was applied on the concrete surface. Four cases of time lag were considered. The number of specimens tested for each case of time lag is shown in Table 7. The same PCM utilized in Section 2.2 was overlaid to the last half after 0, 8, 20 and 44 hours of the surface penetrant application. Before this preliminary experiment, the JSCE-K572 reactive test of the surface penetrant was conducted. The surface penetrant became whiter in two to three days; an invisible reaction must have occurred. Therefore, we set the time lag to 0, 12, 24 and 48 hours. However, the PCM should intensively hydrate in two to four hours after mixing with water. As a result, the actual time lags were adjusted to 0, 8, 20 and 44 hours. Each specimen was cured in a chamber room. The temperature was 20 degrees, and all specimens were wrapped to prevent surface penetrant evaporation, as shown in Fig. 12.

## 4.3 Test results

### 4.3.1 Strength capacity

The average strength of the tested specimens is shown in Table 8. The 0-hour case has a decreased interface strength. In the shear test, two of the three specimens of the 0-hour case were fractured while the jig was being set to these specimens. Thus, 0.33 MPa in the table does not correspond to the average; rather, it is the shear strength of one specimen. Conversely, the tensile test can be performed on the 0-hour case specimens, but the interface strength was smaller than that in the other overlaying time cases. Therefore, applying the surface penetrant right before overlaying PCM is detrimental to the interface strength and causes a significant decrease in the interface tensile strength and shear strength.

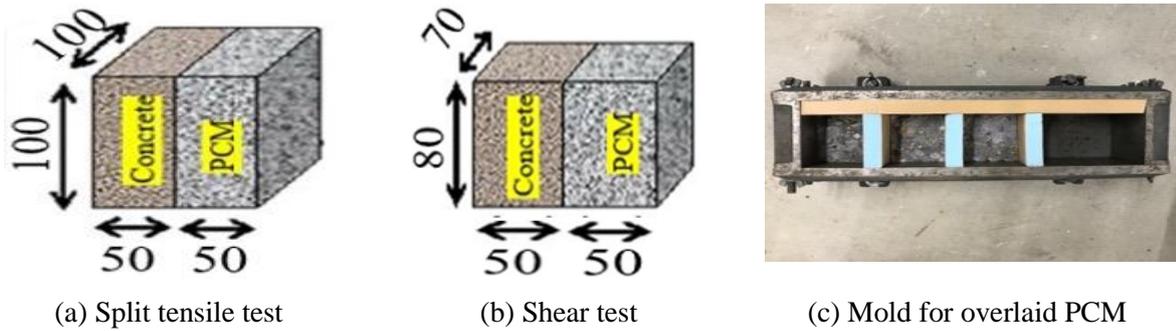


Fig. 11 – Specimen sizes (unit: mm) and mold in the reaction speed experiment



Fig. 12 – Surface curing of the specimen for reaction speed experiment

Table 7 – Number of specimens and types of tests performed

Overlaying time (hours after applying the surface penetrant)	0	8	20	44
Splitting tensile test	3	3	3	3
Shear test	3	3	3	3

Table 8 – Tensile and shear test results

Overlaying time (hours after applying the surface penetrant)	0	8	20	44
Splitting tensile test	0.64	1.41	1.20	1.46
Shear test	0.33*	2.72 (0.63)	2.10 (0.18)	2.08 (0.35)

Note: \* test result of only one specimen; the parentheses value corresponds to standard deviation

For the 8, 20, and 44-hour cases, the results showed stronger interface strength; the results of the tensile test for these cases were approximately two times stronger than the 0-hours cases, and the shear test was more than six times stronger than the 0-hours cases. Therefore, applying surface penetrant with a time lag of at least 8 hours is better to attain interface strength in both tensile and shear test. The 8-hour case showed higher interface strength than the other cases. Thus, a detailed survey of the cases of the delay in the PCM application of approximately 8 hours, with short parameters, may be needed. By reflecting this result to the test strength in Section 3.3,

a stronger interface can be expected because all results shown in Section 3.3 were related to the 0-hour case. For now, the results of Table 6 and Table 8 cannot be compared directly to precisely conclude regarding the application of surface penetrant as different surface roughness preparation techniques were used during specimen preparation of main experiment and reaction time experiment. However, in our further study, same surface roughness techniques will be considered to check the effectivity of the surface penetrant application.

#### 4.3.2 Stress displacement relationship

Fig. 13 shows the relationship between the shear stress and the displacement for different PCM application times. The results of the shear stress displacement curves of the 8-hour case shows the most similar slopes among the three curves. The 8-hour case has the most uniform cases and may yield the strongest interface. Regardless, a delay of more than 8 hours with application of PCM assures its interface bonding.

The stress displacement graphs are less scattered than those of the main experimental results in Section 3.2. This reduced scatter may be attributed to the finding that all fracture modes of the specimens become the same interface fracture mode. In this study, a white surface was also observed. However, the white surface became less white as the time lag increased. When the PCM paste and the surface penetrant are simultaneously applied, the surface

penetrant appears to react with the “Ca ions” of the PCM paste because the PCM that faces the aggregate part also became whiter, and the PCM surface was whiter than the concrete side surface. Therefore, “Ca ions” may be supplied from the PCM paste and be maintained at a fixed concentration. As a result, the surface penetrant can continuously react with “Ca ions” and produce a substantial amount of  $\text{CaCO}_3$ . From the X-rays analysis, 44% of  $\text{CaCO}_3$  was obtained. This amount is considerably greater than usual because calcium hydroxide usually exists at a concentration of approximately 20% in concrete or PCM. As a result, “Ca ions” are considered to be continuously supplied to the interface. In addition, if the surface penetrant made the concrete surface more waterproof, then the PCM could not fill the concrete surface gaps.

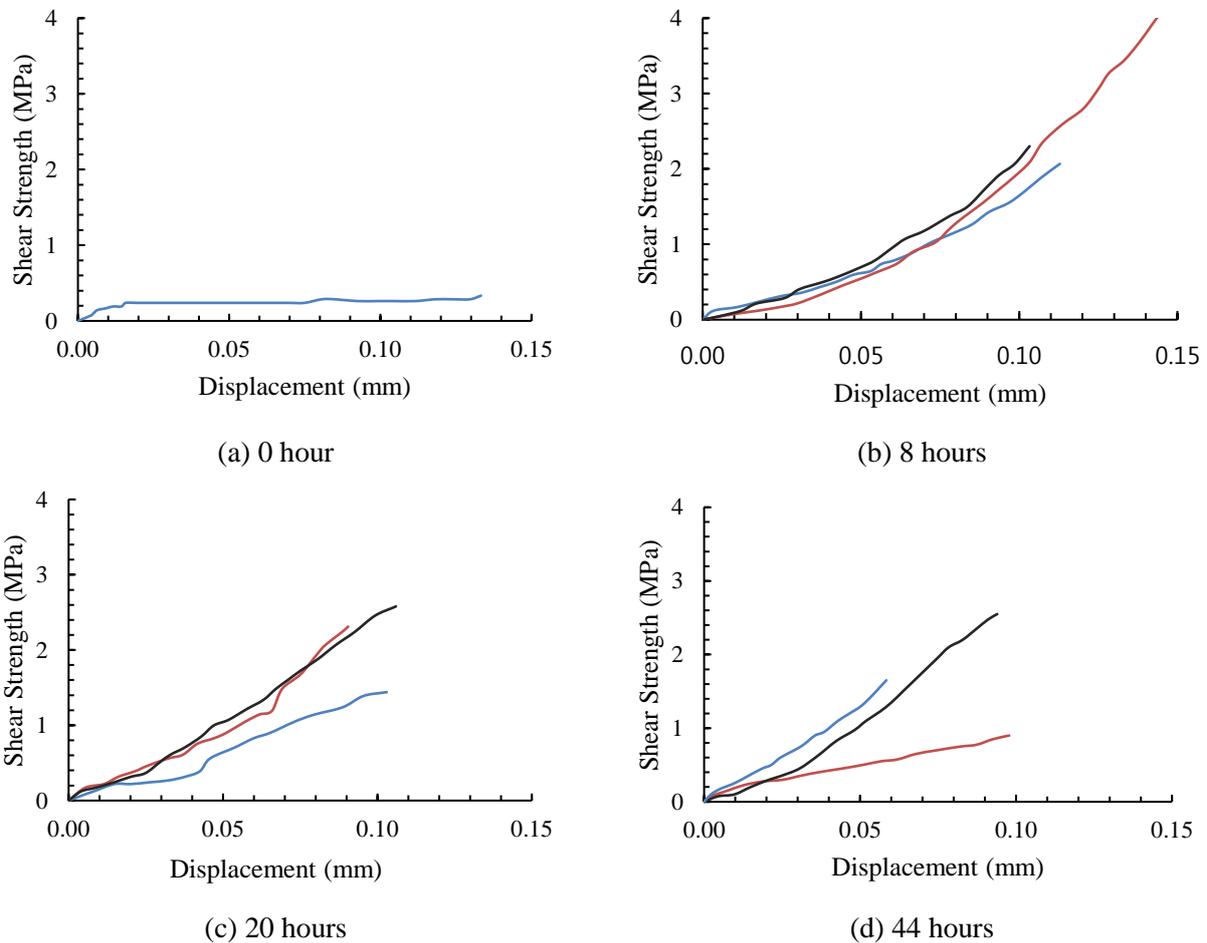


Fig. 13 – Relationship between the stress and the displacement for each time delay case

## 5. Conclusions

The conclusions are summarized as follows:

- 1) By mixing silica fume with PCM, the interface fracture mode shifted from interface adhesion (I) that showed lower fracture energy to concrete cohesion (C-P) showed higher fracture energy.
- 2) Regarding the composite specimens, the silica fume mixed specimens showed stronger interface tensile and shear strength and fracture energy as the amount of mixing of silica fume increases from 0% to 2.5 and 5% of the cement mass.

- 3) Applying the surface penetrant to the interface right before overlaying the PCM causes interface adhesion fracture. Also, it decreases both the tensile and shear interface bonding strengths.
- 4) The weakened interface is attributed to the weak layer of calcium carbonate, which is created by the chemical reaction of the surface penetrant with the fresh PCM at the PCM surface.
- 5) The time lag between the surface penetrant application on the substrate concrete and the PCM application to the substrate concrete should be at least 8 hours to enhance the interface strength.
- 6) Further study is necessary to utilize the interface property enhancement by applying silica fume and surface penetrant for practical cases.

### Acknowledgement

This study was supported by Denka Co., Ltd., who provided many materials (including polymer cement mortar, silica fume, super plasticizer, AE agent and surface penetrant), and Keyence Co., Ltd., who provided access to the roughness measuring apparatus.

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