- Technical Paper -

MECHANICAL RESPONSES OF FRESHLY MIXED CEMENTITIOUS MATERIALS TO SMALL AND SLOW SHEAR DEFORMATION

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ABSTRACT

In this paper a newly developed reference material, which has a high viscosity and contains fine particles, was used to estimate the frictional resistance of the shear box apparatus. Then, the shear box test apparatus was employed to investigate the rheological characteristics of freshly mixed cementitious materials, using shear stress growth method, and shear rate growth method, respectively. A series of shear tests were performed to examine the effects of test method, vertical pressure and specimen's dimension on the mechanical responses of the materials to small and slow shear deformation.
Keywords: cementitious materials, rheological characteristic, influencing factors, reference material, frictional resistance, slow shear deformation

1. INTRODUCTION

In the present rheological studies of fresh concrete, fresh concrete is usually regarded as a homogeneous continuum, and its shear flow behavior is characterized with the Bingham model, which describes the variation of shear rate with shear stress by a linear relationship [1]. However, the Bingham model is unavailable to describe the normal stress-dependence and the thixotropic characteristic of the flow behavior even for high fluidity concrete [2, 3]. Also, when experimental data are fitted in the Bingham model line to determine the Bingham constants of high fluidity concrete, a negative yield stress is often obtained.

For this reason, F. de Larrard et al. proposed to use a non-linear model - the Herschel-Bulkley's model [4]. But when doing numerical simulation, both the Bingham model and the Herschel-Bulkley's model need to be smoothed in order to avoid a sharp angle on the material response to the yield stress. Therefore, an approximation has been usually used to set up the viscosity before yielded to be so large that almost no deformation occurs under a smaller stress less than the yield stress [5]. The rheological characteristics of fresh concrete before yielded, i.e., when it is in small shear rate and deformation, are necessary to be clarified.

At present the rheological performance of fresh concrete is usually measured by several kinds of rotational rheometers, such as parallel plate geometry, coaxial (or concentric) cylinders geometry, and impeller geometry. But there is not yet a generally accepted method [6]. Furthermore, they are mostly shear-rate-controlled methods, and thus it is difficult to measure the behavior in small deformation and when deforming very slowly ($<0.5s^{-1}$). It is considered that stress control test method is possible to measure the mechanical responses of freshly mixed cementitious materials to small and slow shear deformation.

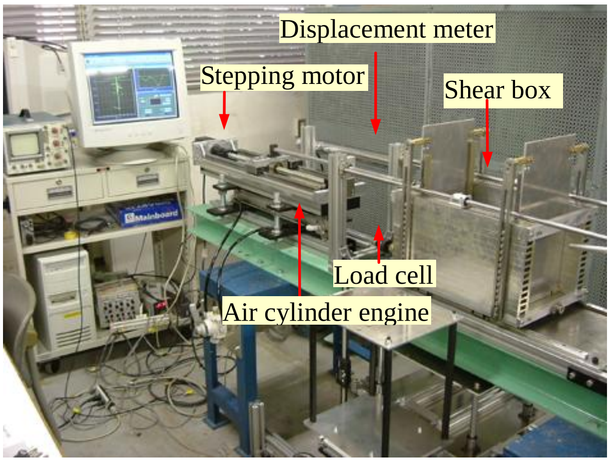

Fig.1 Shear box apparatus for the cementitious materials in fresh state

A shear box apparatus was designed to measure the shear flow behavior of freshly mixed cementitious materials with a shear rate control method, driving by a stepping motor, or with a stress control method, applying arbitrary shear load from zero by a air cylinder engine (see Fig.1) [7]. This apparatus has a potential ability to precisely measure the flow curve of freshly mixed cementitious materials in small shear rate and deformation. But the frictional resistance of this apparatus itself must first be evaluated precisely. For precisely evaluating the resistance, used reference material should have a high and known viscosity, and a density close to that of the cementitious materials.

In this study, a new reference material is developed, consisting of viscous liquid and fine particles. Then the frictional resistance of the shear box test apparatus is measured using this material. After calibrating the apparatus, we measure the flow curves of freshly mixed cementitious materials in small shear

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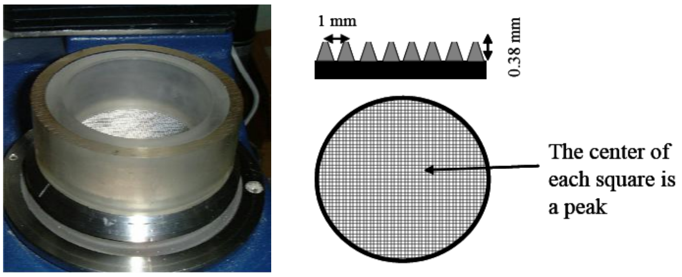

Fig.2 The geometry of the used parallel plate rheometer

rate and deformation with two test methods: shear stress growth method and shear rate growth method. A series of shear tests are performed to clarify the mechanical responses of the cementitious materials to small and low shear deformation, and the effects of test method, normal stress, and specimen's thickness.

2. FRICTIONAL RESISTANCE OF SHEAR BOX TEST APPARATUS

2.1 Reference Material

From some potential candidates of paste, a mixture was selected, which is made of a 75.4% aqueous solution and mineral powder at a 45% particle volume concentration. The solution is with pH 4.48 and a density of 1.387 g/cm^3. The powder is with a density of 2.750 g/cm^3 and a specific surface area of 5500 cm^2/g. And the density of the mixture is 1.989 g/cm^3.

The flow curve of the mixture was measured by a parallel plate rheometer (see Fig.2) [8]. The gap between the two plates was 0.4 mm. The two plates were 35 mm in diameter and were designed in serrated shape to avoid slippage. The peak-to-peak distance is 1 mm, and the depth is 0.38 mm.

The measured flow curve is shown in Fig.3. It is observed that the hysteresis was extremely small and the yield stress was almost zero. Both the up curve and the down curve were nearly linear, thus it is nearly a Newtonian fluid.. The data on the up curve were linearly fitted, as shown in Eq.(1).

$$\tau = 10.32\dot{\gamma} \qquad (1)$$

where τ is shear stress (kPa), and $\dot{\gamma}$ is shear rate (s^{-1}).

Since the mixture has a density close to freshly mixed cementitious materials, high viscosity, and a simple and definite shear stress-shear rate relationship, it is applicable to calibrate the frictional resistance of the shear box apparatus.

2.2 Measurement of the Shear Box Apparatus's Resistance

As shown in Fig.1, the shear box apparatus (box size: 30cm in length, 20cm in width, and 21cm in depth), which was calibrated using the reference material, is constructed by linking several stainless steel frames with linear bearings to reduce the boundary resistance between the specimen and the shear box inside when the specimen deforms. When the top frame or the bottom frame of the shear box is horizontally driven by the stepping motor or the air cylinder engine, two rotating plates revolve in the same direction, and a shear deformation is thus induced in specimen. The vertical pressure is applied by placing a plate-shaped weight on the top surface of specimen.

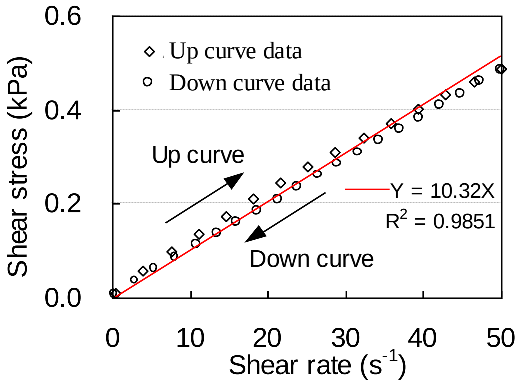

Fig.3 Flow curve of the reference material

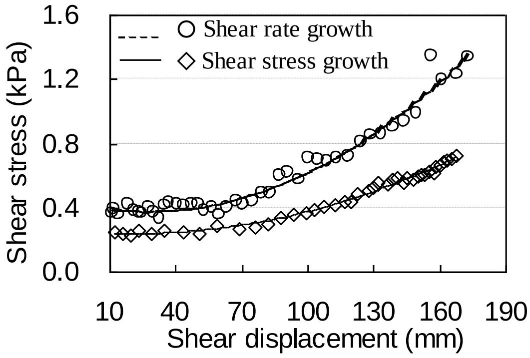

Fig.4 An example of measured shear box apparatus's resistances

2.3 Results and Discussion

The calibrating tests, using the reference material, were performed by a shear rate growth method, and a shear stress growth method, respectively. The effects of specimen thickness and normal stress on apparatus's resistance were also examined. The resistance is a difference between the shear stress measured when the reference material was sheared in the shear box, and the shear stress for only making the reference material deform, calculated by using the Eq.(1). The apparatus's resistance varied with the test method, vertical pressure, specimen's thickness, etc. Fig.4 shows an example of test results under the conditions of specimen thickness of 15cm, different test methods: shear rate growth method (normal stress was 0.67 kPa) and stress growth method (normal stress was1.33 kPa).

The apparatus's resistance models under different conditions are shown in Table 1. Due to the inertia force at the beginning of driving the shear box, the load and the resistance sharply fluctuated in the shear displacement range of 0 ~ 10mm. Hence, the models apply to the displacement range of 10 ~ 180 mm. The coefficients of determination are high, thus the models shown in Table 1 have satisfactory precision.

Table 1 The resistance models of the shear box apparatus

	Specimen thickness (cm)	Normal stress (kPa)	Resistance models	Coefficient of determination (R^2)
Shear Stress Growth Type (Rate of loading: 5N/s)	15	0	$F_r = 0.022x^2 - 3.220x + 177.9$ $10\text{mm} < x < 180\text{mm}$	0.935
		0.33	$F_r = 0.022x^2 - 2.462x + 246.8$ $10\text{mm} < x < 180\text{mm}$	0.972
		0.67	$F_r = 0.028x^2 - 3.850x + 258.1$ $10\text{mm} < x < 180\text{mm}$	0.933
		1.33	$F_r = 0.022x^2 - 1.014x + 251.9$ $10\text{mm} < x < 180\text{mm}$	0.989
	21	0	$F_r = 0.030x^2 - 3.357x + 265.3$ $10\text{mm} < x < 180\text{mm}$	0.968
Shear rate Growth Type (Max. displacement rate: 6 cm/s)	15	0	$F_r = 0.039x^2 + 0.281x + 440.2$ $10\text{mm} < x < 180\text{mm}$	0.961
		0.33	$F_r = 0.011x^2 + 0.444x + 615.6$ $10\text{mm} < x < 180\text{mm}$	0.910
		0.67	$F_r = 0.045x^2 - 2.504x + 406.8$ $10\text{mm} < x < 180\text{mm}$	0.970
		1.33	$F_r = -0.031x^2 + 12.67x + 257.9$ $10\text{mm} < x < 180\text{mm}$	0.964
	21	0	$F_r = 0.039x^2 + 0.312x + 439.7$ $10\text{mm} < x < 180\text{mm}$	0.961

[Notes] F_r is the frictional resistance of the shear box (kPa), and x is shear displacement (mm).

Table 2 Mix proportions and the properties of the used concretes

Series	Mix Proportions		Unit Mass (kg/m^3)					Properties in Fresh State			
	W/C (%)	*S/a* (%)	*W*	*C*	*S*	*G*	*Sp* (*C*×%)	*Sl.* (cm)	*Sf.* (mm)	T_{50} (s)	ρ (g/cm^3)
C1	50	37	175	350	657	1119	1.5	-	603	3.65	2.413
C2	50	42	170	340	753	1040	1.0	21.0	-	-	2.319
C3	50	38	170	340	674	1100	1.0	14.0	-	-	2.340

[Notes] *W/C:* Water-cement ratio; *S/a:* Sand-aggregate ratio by mass; *W, C, S* and *G:* Water, cement, sea sand, and crushed stone, respectively; *Sp:* Air entrained super-plasticizer; *Sl.:* Slump; T_{50}: 50cm-flow time; *Sf.:* Slump flow, and ρ: Unit mass of concrete.

Table 3 Mix proportions of the used mortars

Series	M1	M4	M5	M6
W/C (%)	30	40	45	50
S/C	1.20	1.50		

[Notes] *W/C:* Water-cement ratio, and *S/C:* Sand-cement ratio by mass.

3. EXPERIMENTAL INVESTIGATION ON FLOW BEHAVIORS OF CEMENTITIOUS MATERIALS

3.1 Raw Materials

Mix proportions of used concretes and mortars are shown in Tables 2 and 3. And there were two kinds of cement pastes, whose water-cement ratios (W/C) were 0.30 and 0.35, and named CP-1 and CP-2, respectively. For producing these cementitious materials, ordinary portland cement was used, with a specific gravity of 3.16 g/cm^3. The sea sand was used as fine aggregate, of which the saturated surface dry density is 2.59 g/cm^3, the coefficient of water absorption is 1.60%, and the fineness modulus is 2.57, respectively. Continuously graded crushed sandstone, with the saturated surface dry density of 2.73 g/cm^3, the coefficient of water absorption of 0.40%, the maximum size of 20 mm, and the fineness modulus of 6.67, was used in the concretes as coarse aggregate. Standard type air-entraining (AE) super-plasticizer was also used, of which dosage was in the percentage of cement by mass.

3.2 Test Procedure

As the test procedure, right after the mixture (concrete, mortar, cement paste) was mixed, it was packed into a polyethylene bag that was previously treated with lubricant, and placed into the shear box. When examining the influence of normal stress, a plate- shaped weight was placed on the top surface of the specimen.

If testing with the shear stress growth method, the horizontal force was increased gradually from zero by the air cylinder engine. Meanwhile, the shear force and the horizontal displacement were measured with a load cell. When the displacement reached 180 mm, the test was over. In case of testing with the shear rate growth method, the shear box was driven by the stepping motor at a certain acceleration until the shear displacement rate reached a certain value set up beforehand.

Based on the test results of time-load-displacement relationship, shear stress, shear strain, and the shear rate were calculated respectively. Then, the shear strain -shear stress relational curves and the shear rate-shear stress relational curves were further plotted for the tested mixtures.

3.3 Results and Discussion

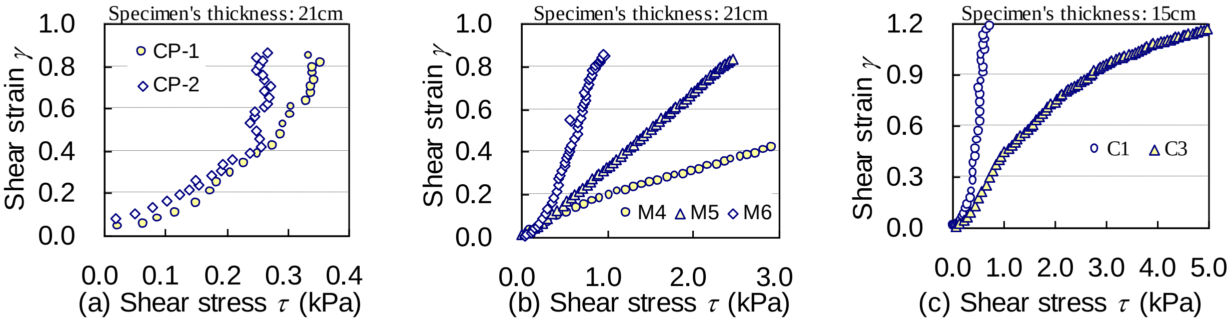

Fig.5 Shear strain – shear stress relationship measured with shear stress growth method

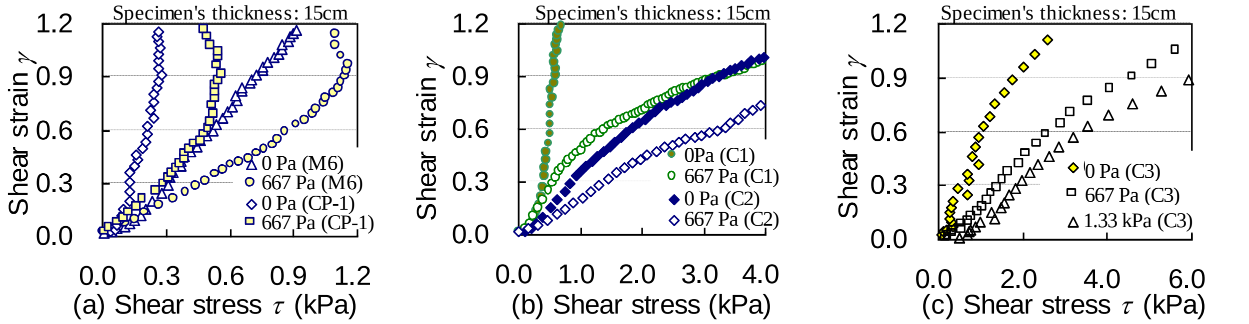

Fig.6 Effect of normal stress on the $\gamma-\tau$ relationship ((a), (b): Stress growth method, (c): Shear rate growth method)

(1) Shear strain -shear stress relationship

Fig.5 shows the shear strain-shear stress relationships for three kinds of freshly mixed cementitious materials under the vertical pressure of 0 Pa., measured with the shear stress growth method. In order to clearly clarify the behaviors in small shear deformation region, the shear strain was limited to a range of 0~1.2. As can be seen from this figure, the shear strains of all the specimens increased with the applied shear stress. In the early stage with very small shear stain, the increases were rapid, the curves were convex down (hereafter called briefly this stage as "rapid increase satge"), but after a cetrain shear stain, the increases became slow, thus, the curves were convex up (hereafter called briefly this stage as "slow increase satge"). The "lengths" of the rapid increase satge were in order of cement paste, mortar and concrete. The rapid increase satge of the cement pastes were so long that the slow increase stage didn't be found within the measuring range of shear strain. For the same kind of specimen, the lower the fluidity, the shorter the rapid increase stage. These trends were also found from the tests with the shear rate growth method (see Fig.6 (c)).

This is probaly because the particles in the freshly mixed cementtitious materials don't contact closely, are in a suspended state in the initial stage, thus the liquid feature of fresh cementitious materials was dominant. However, as the deformation of the specimen growed, the contact between the particles became closer and closer, the dilatancy took place easily. The resistance resulting from the dilatancy would reduce the increase of the shear strain with shear stress. Whereas, the cement pastes had lower solid particle concentrations, thus, a dilatancy hardly occured with the increase of shear deformation, the resistance casued by the dilatancy was small.

Morever, with the deformation increasing, the angle between the rotating plate and the bottom plate of the shear box becomes smaller and smaller, which would yield a higher restraint of container to the specimen with a higher particle concentration or containing coarser particles. This maybe is another reason why the concretes had a smaller increasing rate of shear strain.

Fig.6 shows the shear strain-stress relationships for three kinds of materials, measured with the two test methods under different normal stresses. The varying trend of the measured shear strain with the shear stress was the

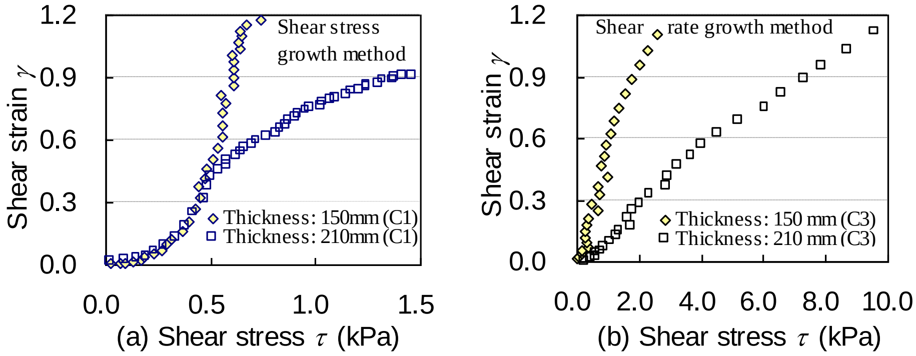

Fig.7 Effect of specimen's thickness on the $\gamma-\tau$ relationship of fresh concrete

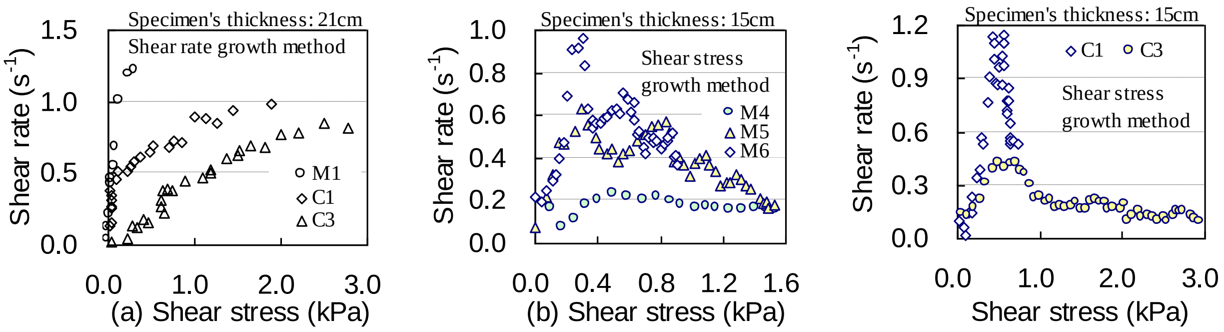

Fig.8 Flow curves of different mixtures, measured with different measurement methods

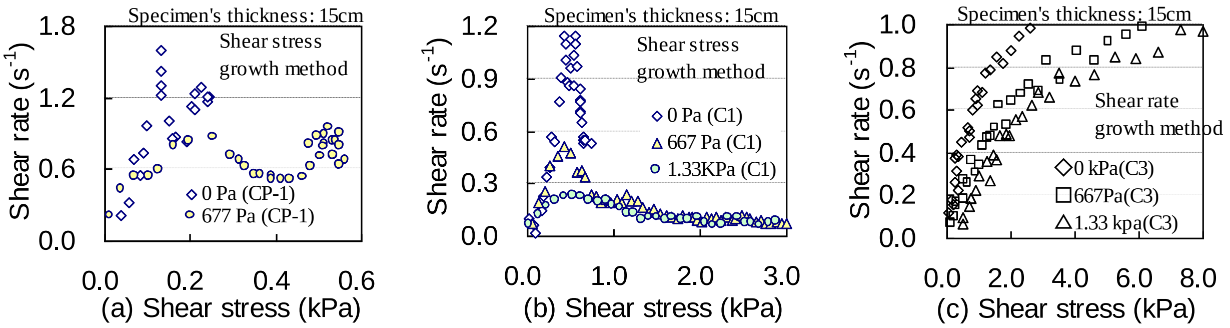

Fig.9 Flow curves of the mixtures under different normal stresses, measured with the different measurement methods

same for the two test methods. Also, for no matter what kind of material, with increasing the normal stress, the shear strain decreased. Also, with increasing the normal stress, the "length" of the rapid increase stage became short. It is considered that the effect of normal stress is associated with the particle contact point angles that result in a dilatancy-caused resistance. With increasing the normal stress, the inter-frictional resistance and the dilatancy-caused resistance will increase [9].

Fig.7 shows the effect of specimen's thickness on the shear strain-shear stress relationships of fresh concretes for different shear layer thickness, measured with the different test methods. For either of the concretes and the test methods, the shear strain decreased with the increase of the specimen's thickness. This dimension's effect is considered to result from that the greater the specimen's thickness, the larger the normal stress caused by its gravity. Greater normal stress would result in a larger dilatancy-caused resistance.

Because the density of concrete is greater than that of the reference material, more underestimation of the actual resistance of the shear box filled by concrete of 21 cm than 15 cm in depth is probably another reason.

(2) Shear rate -shear stress relationship

Fig.8 (a) shows the flow curves for three kinds of the cementitious materials measured with the shear rate growth method, while the flow curves shown in Fig.8 (b) and (c) were measured with the shear stress growth method. During the measurements, no vertical pressure was applied. When measured with the shear rate growth method, the shear displacement rate was limited to a small range of 0~6 cm/s at certain acceleration for clearly clarifying the flow performance in a low shear rate state. As shown in Fig.8 (a), the shear rate increased with increasing the shear stress. All the flow curves were non-linear. The flow curves also showed the rapid increase stage and the slow increase stage.

The slow increase stage is considered as shear-thickening phenomenon. When fresh mortar or concrete flows rapidly or deforms in large, the concentrated particles tend to disperse, thus the particle inter-locking and the dilatancy are hard to take place. However, if fresh concrete or mortar is in a small deformation or deforms at a low speed, it is in a visco-elsto-plastic state. Thus, the dilatancy-caused resistance increases with increasing the shear stress, because the mean particle contact point angle that associates with the dilatancy-caused resistance increases with the shear stress [10]. The increase in the dilatancy-caused resistance would reduce the

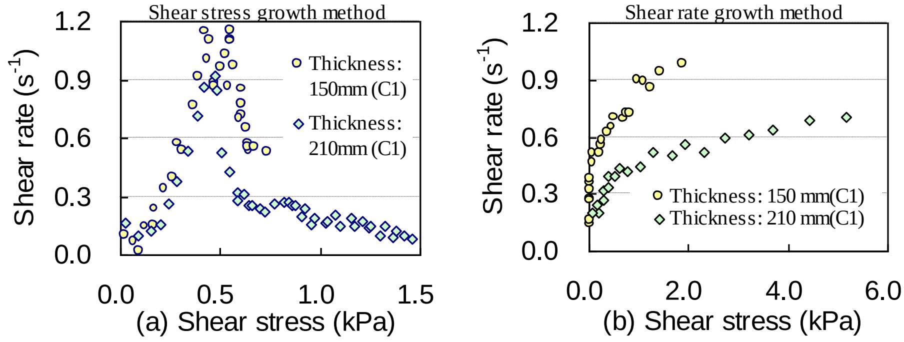

Fig.10 Flow curves of fresh concretes under different specimen's thicknesses

increasing rate of shear rate.

On the other hand, in case of testing with the shear stress growth method (load speed was 5 N/s), unusual test results were obtained that the shear rate increased with applied shear stress in the initial stage (increase stage), then decreased with increasing the shear stress after reaching a peak shear rate (decrease stage). The lower the fluidity of the specimen, the smaller the shear rate. We consider that in the initial stage with extremely small deformation, the materials show a liquid feature as stated above, after a certain shear deformation, the materials show a granular feature. When using the stress growth method, the shear stress was increased under compulsion, and the shear deformation occurred responding to the applied shear stress, but the shear rate was unrestricted. As mentioned above, in case of small deformation, due to the effect of the shear dilatancy, the shear deformation would become slow, though the final deformation reaches to a certain value answering to the applied shear stress. Because of the dilatancy effect, the shear rate was very small even for a greater shear stress. If the shear stress is further increased greatly, the shear rate will increase with shear stress. This phenomenon was observed from the test results of the cement pastes and M6 (see Fig.8 (b) and Fig.9 (a)). Unfortunately we didn't find it from the concrete's experiments due to the limited shear deformation of the used shear box. We consider this phenomenon to be a behavior in the shear failure state, and will discuss this behavior in other paper in detail.

Fig.9 indicates the flow curves of different cementitious materials under different normal stress levels, measured with the shear rate growth method. For any kinds of the specimens, the shear rate decreased with increasing the normal stress.

Fig.10 indicates the flow curves of the fresh concrete with different shear layer thickness, measured with the two methods. The thicker the specimen, the smaller the shear rate for any of the two kinds of the test methods. This is also considered to be attributed to the change of the normal stress caused by the specimen's gravity.

4. CONCLUSIONS

In this study, the mechanical responses of freshly mixed cementitious materials to small and slow shear deformation were investigated. The main conclusions are as follows:

(1) The fractional resistances of the shear box apparatus under different conditions were examined with the mixture, made of a 75.4% aqueous solution and inorganic mineral powder at a 45% particle volume concentration. The frictional resistance models were obtained as given in the Table 1.

(2) The trends of flow curves of freshly mixed cementitious materials were different from the test methods. The shear rate increased with increasing the shear stress, when using the shear rate growth method. However, when using the shear stress growth method, the shear rate firstly increased with shear stress, then decreased with the shear stress after a peak shear rate.

(3) The shear strain of fresh concrete increased with the shear stress. The increasing rate of the shear strain increased with the shear stress in the early stage, then decreased with the shear stress.

(4) The flow behaviour of fresh concrete is depend on the normal stress on the shear plane and the specimen's dimension. With increasing the normal stress or the specimen's thickness, the shear strain and the shear rate decreased no matter which test method is used.

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