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Improvement of autogenous shrinkage measurement for cement paste at very early age: Corrugated tube method using non-contact sensors

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HIGHLIGHTS

- Test methods for autogenous shrinkage of cement paste are reviewed.
- Few methods are able to measure the very early autogenous shrinkage of cement paste.
- A non-contact corrugated tube system was designed.
- Results obtained by the designed system show superior accuracy and repeatability.

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ABSTRACT

Test methods for the measurement of autogenous shrinkage of cement paste were reviewed, and it is found that few methods can precisely determine the autogenous shrinkage of cement paste at very early age. Therefore, the corrugated tube method was improved by using non-contact linear variable differential transformer (LVDT) sensor for measuring the autogenous shrinkage of cement paste at very early age. The experimental results show that the transformation ratio of the volume deformation to length change for the designed non-contact system can be as high as 0.97, which gives a significant improvement compared to that for the contact system (0.87). Compared to the contact system, the designed non-contact system also has an improved repeatability: the typical standard deviations reach about 50 μm/m and 10 μm/m before and after setting, respectively.

1. Introduction

1.1. Autogenous shrinkage

Autogenous shrinkage is generally defined as "the unrestrained, bulk deformation that occurs when cement-based materials are sealed and no loss of moisture or mass occurs with the external environment under a constant temperature" [1]. It mainly attributes to the chemical shrinkage and self-desiccation shrinkage. Chemical shrinkage, also known as Le Chatelier's contraction, is a continuous feature of cement-based materials during the hydration process, since hydration products occupy less absolute volume than reactants (water and cement) [1,2]. When stiff skeleton structure is formed in cement paste during the hydration process, internal voids form in the matrix due to chemical shrinkage, leading to the formation of water–air menisci and continuous reduction of the internal relative humidity (RH) [3,4]. Consequently, tensile stress is generated in the pore solution, resulting in the self-desiccation shrinkage [5].

Chemical shrinkage is an absolute volume change (internal), and autogenous shrinkage is an apparent volume change (external) [2,6]. The schematic diagrams of chemical shrinkage and self-desiccation shrinkage are presented in Fig. 1. Even hydration of only one cement particle can result in chemical shrinkage (Fig. 1(a)). Before the formation of stiff skeleton structure, autogenous shrinkage of cement paste is equal to the sum of chemical shrinkage of individual cement particle. Self-desiccation shrinkage is an apparent shrinkage and cannot exist without the formation of voids in cement paste (Fig. 1(b)). After the formation of stiff skeleton structure, self-desiccation shrinkage is considered as the main part of autogenous shrinkage. During the two periods of hydration process, chemical shrinkage is always a fundamental factor of autogenous shrinkage.

Autogenous shrinkage is increasingly concerned for the sake of maintaining durable civil engineering buildings and constructions using cement-based materials with a low water to cement ratio (w/c) and can be divided into two parts: shrinkage at early age (the first 24 h) and shrinkage in long term (after 24 h) [7]. The former, accounting for the main proportion of ultimate shrinkage, is the main factor inducing the cracking of cement-based constructions [8,9]. Therefore, the investigation on the autogenous

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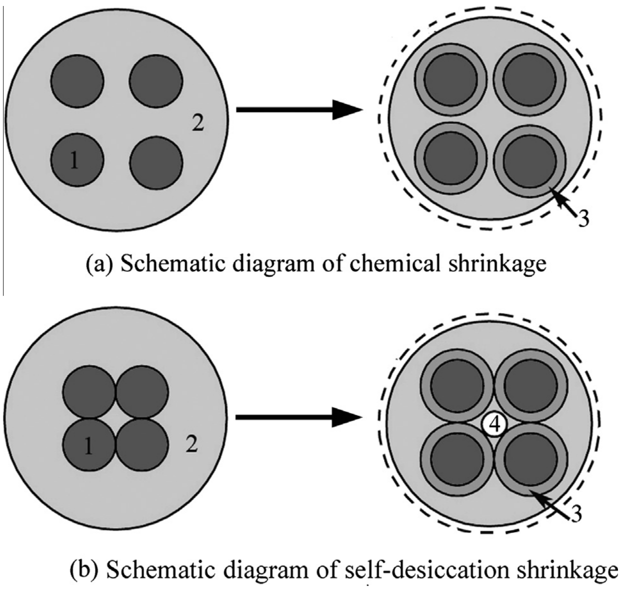

Fig. 1. Schematic diagram of chemical shrinkage and self-desiccation shrinkage. (1) Anhydrous cement, (2) pore solution, (3) hydration products, and (4) air void.

shrinkage of cement-based materials at early age is very important [10]. However, the precise measurement is the major problem as reported [11]. Following is the literature review on the measurement methods for autogenous shrinkage of cement paste and especially on the measurement at very early age.

1.2. Test methods for autogenous shrinkage of cement paste

Measurement methods for determining the autogenous shrinkage of cement paste can be classified into volumetric and linear methods [4,7,12,13], as shown in Fig. 2. In the volumetric method, cement paste is introduced into a membrane (condom was normally used) and cured under isothermal water (Fig. 2(a)). The volume change of external water or buoyancy change of cement paste is recorded to represent the autogenous shrinkage. The main advantage of this method is that it can measure the initial autogenous shrinkage of cement paste immediately after mixing [13]. However, its precision is influenced by many factors, e.g. the bleeding effect and the membrane permeability. The bleeding water accumulated on the surface of cement paste before setting can be re-absorbed in the sample, causing additional shrinkage [13,14]. Bouasker et al. [11] proposed a dynamic system by rotating the sample during the whole testing period to eliminate the bleeding effect. However, the fluctuation of the measured data was also resulted from the rotation. In terms of the influence of membrane permeability, water can continuously transport into the pore solution of cement paste through the membrane after immersing, increasing the buoyancy of cement paste and causing a significant error. Lura and Jensen [15] reduced this error by immersing the sample into paraffin oil instead of water. However, Bouasker et al. [16] indicated that the error cannot be perfectly eliminated, because paraffin oil can be absorbed on the membrane. Additionally, the volumetric method is not suitable to measure the autogenous shrinkage for concrete, because the membrane can be easily broken by the sharp aggregate.

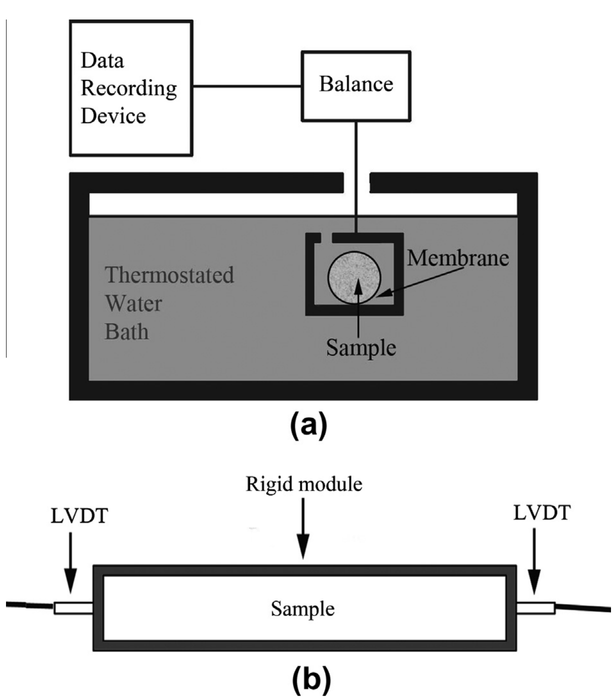

Fig. 2. Typical test methods for the autogenous shrinkage of cement paste. (a) Volumetric method, and (b) linear method.

In the linear method, cement paste is commonly cast in a rigid module with a low friction (Fig. 2(b)) [10,11,17,18]. This method can also be classified into two categories: horizontal and vertical methods. In both methods, displacement sensors, e.g. linear variable differential transformers (LVDT) are commonly used to record the length change of cement paste. The main shortage of the horizontal method is that it cannot precisely measure the autogenous shrinkage of cement paste at plastic stage, because the length change cannot represent the volume deformation during this period [13]. In vertical method, although the autogenous shrinkage can be immediately measured after casting, the error also occurs due to the influence of gravity force [14].

Jensen and Hansen [12] proposed a linear method to determine the autogenous shrinkage of cement paste at early age by using corrugated tubes, as shown in Fig. 3. The corrugated tubes transform the volume deformation into length change before the formation of stiff skeleton structure, which solves the main drawback of the previous reported linear methods. This method was widely used to investigate the early age autogenous shrinkage of cement-based materials [13,15,16], and a standard test method for autogenous strain of cement paste and mortar in terms of corrugated tube method was published by American Society for Testing Materials [19].

However, considerable errors are caused by the stress between the contact sensors and corrugated tube before the formation of

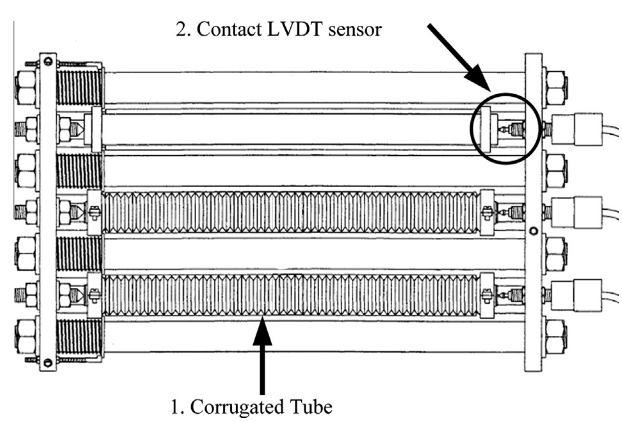

Fig. 3. Contact corrugated tube method proposed by Jensen and Hansen [12].

stiff skeleton structure and cannot be eliminated due to the characteristic of contact sensors, because the stress is needed to obtain the output of contact sensors. Consequently, the contact systems are commonly applied for the measurement of autogenous shrinkage of cement paste after setting.

2. Improvement of corrugated tube method

In order to increase the measurement precision for the autogenous shrinkage of cement paste at very early age, the corrugated tube system was improved by using non-contact LVDT sensors in this study. The principle of designed non-contact system is simplified as follows: cement paste is firstly cast into the corrugated tube, and then the volume deformation of cement paste is converted into the linear change of corrugated tube which can be transformed into voltage signals by non-contact LVDT sensor. At the same time, the voltage signals are recorded automatically by the computer.

As shown in Fig. 4, the non-contact corrugated tube system consists of: (1) Non-contact LVDT sensor, (2) Conditioning module, (3) Analog to digital converter, (4) DC regulated power supply, (5) Computer, (6) Methylmethacrylate module used to support corrugated tubes and sensors; (7) Corrugated tubes made by low density polyethylene (LDPE).

2.1. Introduction of the designed non-contact system

2.1.1. Non-contact LVDT sensor

There are three wires in sensor tube: One is a primary cycle, and the other two are connected together, called secondary cycle. The induction electromotive forces between the primary and secondary cycle are equal when the inner magnetism bar locates at the midpoint of sensor tube, and the induction electromotive forces are different when the inner magnetism alters its initial position, leading to a voltage output of the sensor. It should be noted that the displacement of inner magnetism bar is directly proportional to the voltage output of sensor. The conditioning module is used to magnify the voltage output of sensor in order to record the data easily. The non-contact sensor and conditioning module are combined in a displacement measurement system with the following parameters: voltage output ranging from −5 to 5 V, corresponding displacement output ranging from −6.35 to 6.35 mm and working temperature ranging from −55 to 150 °C. Therefore, the relationship between the displacement and voltage output is expressed as Eq. (1), and the fitted line is given in Fig. 5.

$$S = (6.35/5) \times U \tag{1}$$

where S (mm) is the displacement, U (V) is the voltage output.

2.1.2. Analog to digital converter

The displacement resolution of designed non-contact system depends on the analog to digital converter. Because the voltage output resolution of the analog to digital converter (16 bits) is 0.15 mv, the corresponding displacement resolution is 0.19 μm according to Eq. (1), which meets the requirement of ASTM C1698-09 [19].

2.1.3. Corrugated tube and plug

Corrugated tube is made of LDPE by the method of injection blowing modeling method. Tian and Jensen [13] indicated that the corrugated tube made of LDPE is more flexible to maintain a higher transformation ratio of volume deformation to length change (defined in 2.2) that made of high density polyethylene (HDPE). The lengths of the whole tube, corrugated part, inner and outer diameters are 280 mm, 250 mm, 25 mm, and 36 mm, respectively. The plug is made of polyformaldehyde.

2.2. Verification of transformation ratio (volume deformation to length change ratio)

Transformation ratio of volume deformation to length change (abbreviated as transformation ratio) is defined as the volume deformation of cement paste in corrugated tube to the length change of corrugated tube ratio. In theory, the volume deformation of cement paste in corrugated tube is assumed to be equal to the

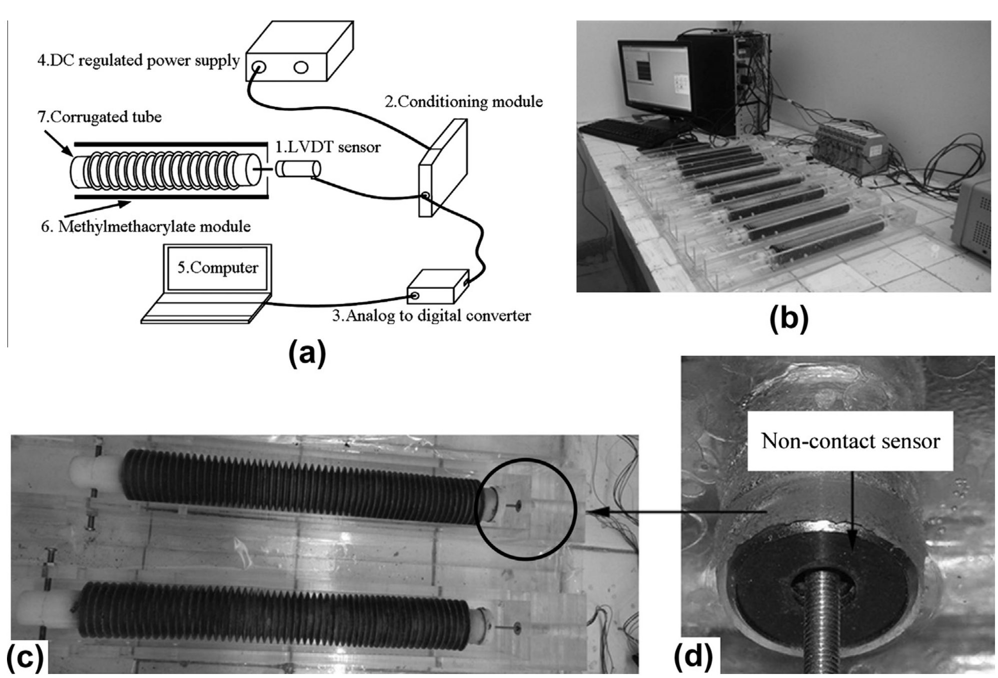

Fig. 4. The designed system for autogenous shrinkage of cement paste using non-contact LVDT sensor: (a) schematic diagram of the designed system, (b) photograph of the system, (c) corrugated tubes, and (d) non-contact sensor.

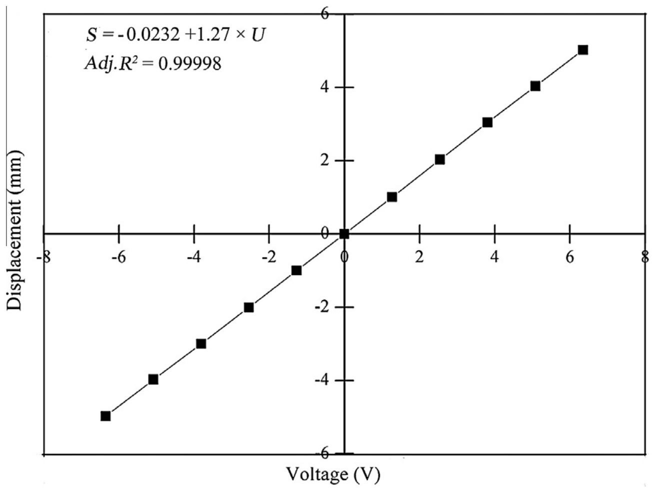

Fig. 5. Typical LVDT calibration curve.

volume deformation of the corrugated tube, and can be totally transformed into the length change of corrugated tube. Therefore, the transformation ratio is equal to 1.00 [10,13]. In the real measurement, however, due to the stiffness of corrugated tube, and the friction between corrugated tube and supported module, the volume deformation of cement paste in corrugated tube is not totally transformed into the length change of corrugated tube. As a result, the transformation ratio is lower than 1.00 [13].

Obviously, the precision of corrugated tube measurement can be improved by increasing the transformation ratio. Although the stiffness of corrugated tube is often measured to illustrate its flexibility [10,13,16], verifying the transformation ratio of volume deformation to length change is more important. However, rarely literature reported this kind of verification. The main reason may be that it is difficult to directly determine the volume deformation of cement paste in corrugated tube due to the continuous hydrating of cement.

Therefore, in order to verify the transformation ratio, Tian and Jensen [13] tested the volume deformation by using distilled water. They found that the length change of corrugated tube is directly proportional to the volume deformation of distilled water in the corrugated tube, and the transformation ratio can be calculated by Eq. (2):

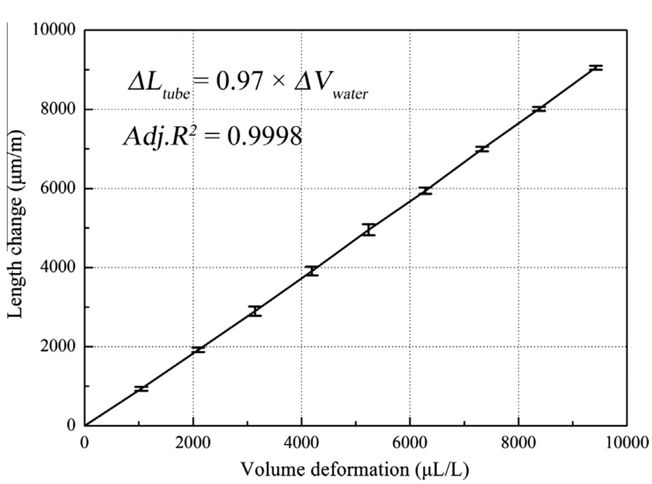

Fig. 6. The relationship between length change and volume deformation of corrugated tube.

$$k = \frac{\Delta L_{tube}}{\Delta V_{water}} \tag{2}$$

where ΔL_{tube} is the length change of corrugated tube, ΔV_{water} is the volume deformation of materials in corrugated tube, k is the transformation ratio.

Based on Tian and Jensen's method [13], the transformation ratio of volume deformation to length change was verified in this study. The non-contact corrugated tube measurement system was placed at the isothermal room (20 ± 1 °C). The distilled water (previously placed at the isothermal room for 1 day) was firstly injected into the corrugated tube with a constant length of 250 mm. The corrugated tube was sealed with plastic plugs and epoxy adhesive. Certain amount (1, 2, 3, 4, 5, 6, 7, 8, 9, 10 mL, respectively) of distilled water in the corrugated tube was absorbed by an injector (20 mL) to represent the volume deformation of corrugated tube. Accordingly, the voltage output of the sensor was monitored to calculate the length change of the corrugated tube. The relationship between the volume deformation and the length change of corrugated tube is shown in Fig. 6 (the results were the average value of three verifications). According to Eq. (2), the transformation ratio is the slope of fitted line in Fig. 6.

The transformation ratio of the designed non-contact system (0.97) is lower than 1.00 (Fig. 6), indicating that the volume deformation is not totally transformed into the length change. It may be related to the friction between the corrugated tube and the supported module. However, compared to the reported results (0.87) of Tian and Jensen's contact system [13], the transformation ratio of the designed non-contact system (0.97) is significantly improved by 0.10. The main reason is that the stress between the sensor and corrugated tube is eliminated in this system.

3. Experiment and validation

3.1. Materials

The cement paste was prepared with type I 42.5 Portland cement and its chemical composition is given in Table 1. The Blain fineness is 367 m^2/kg, and specific gravity is 3.14 g/cm^3. The Bogue mineral composition is: C_3S 56.20%; C_2S 19.63%; C_3A 6.54%; C_4AF 8.97%; $CaSO_4 \cdot 2H_2O$ 3.5%. Distilled water with specific gravity of 1 g/cm^3 was used as the mixing water. Three w/c ratios (0.26 (the water requirement of normal consistency, determined according to European standard EN 196-3 [20]), 0.30 and 0.40) were used to investigate the influence of w/c ratios on the autogenous shrinkage of cement paste.

3.2. Setting time

The Vicat initial and final setting times were determined according to European standards EN 196-3 [20]. Table 2 provides the Vicat setting times of cement pastes with different w/c ratios. The setting time increased with the increase of w/c ratio, because the cement paste with a higher w/c ratio has a larger initial porosity, leading to a longer setting time [5].

3.3. Autogenous shrinkage of cement paste

Cement paste was cast according to EN 196-3 [20]. The corrugated tube was primarily kept with a constant length of approximate 250 mm, and the cement paste was injected into the tube with the aid of table vibrator to eliminate the air void in the tube. The tube was then sealed with the plastic plug. The data was initially recorded at 30 min after mixing and then logged per 1 min during the test. The autogenous shrinkage was calculated by Eq. (3):

$$\varepsilon_{AS} = (S_t/L_0) \times 100\% \tag{3}$$

Table 1
Chemical composition of the Portland cement.

Chemical composition (wt%)									
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	LOI	Others
21.60	4.35	2.95	63.81	1.76	0.51	0.16	2.06	1.19	1.61

Note: LOI, loss on ignition.

Table 2
Vicat setting times of cement pastes.

w/c Ratio	Initial setting time (min)	Final setting time (min)
0.26	102	172
0.30	145	191
0.40	216	276

where ε_{AS} (μm/m) is the autogenous shrinkage at time t, S_t is the displacement of tube at time t, L_0 is the initial true length of paste.

3.4. Repeatability of the designed non-contact system

The repeatability of the designed non-contact system was verified by three parallel samples (designated as 1, 2, 3, respectively) with the w/c ratio of 0.26. The standard deviation of recorded data of above three samples was used as a parameter to illustrate the repeatability of the designed non-contact system. The standard deviation was calculated by Eq. (4):

$$s = \sqrt{(1/(N-1)) \times \sum_{i=1}^{N}(x_i - \bar{x})^2} \tag{4}$$

where s is the standard deviation, x_i is the value of sample i, $\bar{x}$ is the average value of all samples, N is the number of samples.

The repeatability of the present autogenous shrinkage measurement is shown in Fig. 7. The recorded autogenous shrinkages values of the three samples exhibited a higher difference during the first 12 h, and the recorded values were almost identical after 12 h. The standard deviation significantly increased after mixing and reached the maximum value of approximate 50 μm/m around the initial setting time (110 min), and then reduced and maintained at about 10 μm/m (Fig. 7(b)).

The recorded autogenous shrinkages of cement paste at plastic status were more sensitive to the stress, friction, void and other artifacts, thus the standard deviation of recorded data was higher at very early age than that at later age [16]. The standard deviation of autogenous shrinkage data were rarely reported except that in Jensen and Hansen's work [11], a typical standard deviation value of 200 μm/m and 20 μm/m was calculated for the contact measurement before and after setting, respectively. Compared to this value, the standard deviation of the present non-contact measurement system was reduced by 150 μm/m and 10 μm/m before and after setting, respectively, indicating that a significant improvement of the repeatability. The improvement is related to no stress between the non-contact sensors and corrugated tube at very early ages.

3.5. Comparison between the designed non-contact system and other non-contact system

The autogenous shrinkages of cement pastes with different w/c ratios prior 24 h are presented in Fig. 8(a). The autogenous shrinkage of cement paste with a w/c ratio of 0.26 was significantly larger than others during the whole testing period: after 24 h, the autogenous shrinkage of cement paste with the w/c of 0.26, 0.3 and 0.4 is around 1400 μm/m, 850 μm/m and 400 μm/m, respectively. Bouasker et al. [16] used the non-contact (eddy-current sensors) corrugated tube method to measure the autogenous shrinkage of cement paste. Similarly with the present results, the

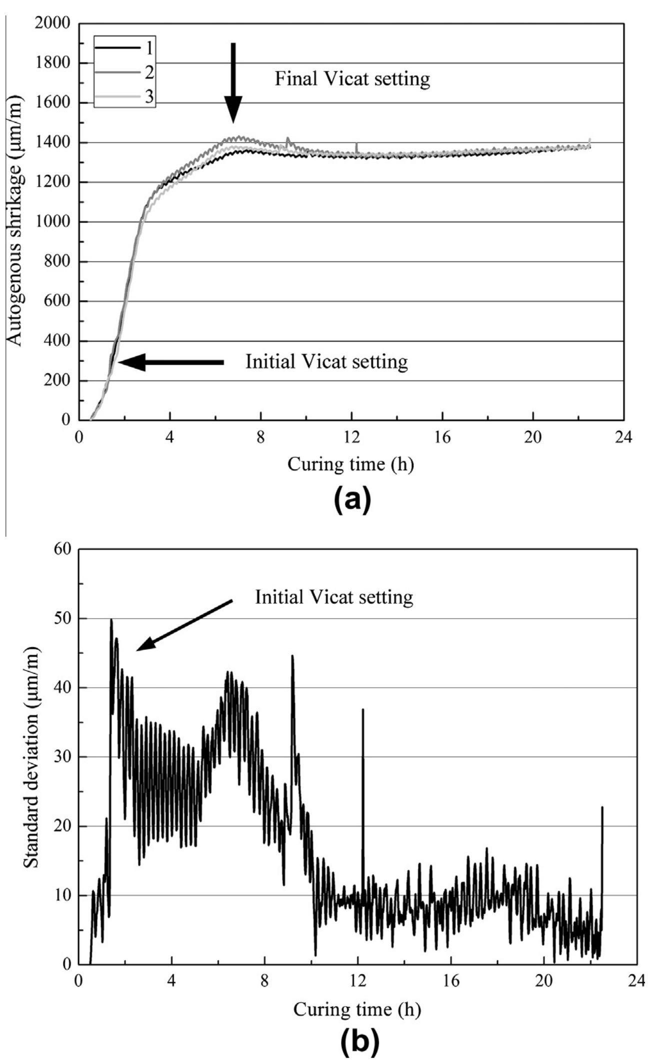

Fig. 7. The repeatability of the present autogenous shrinkage measurement. (a) Autogenous shrinkage versus curing time, and (b) standard deviation of the obtained results.

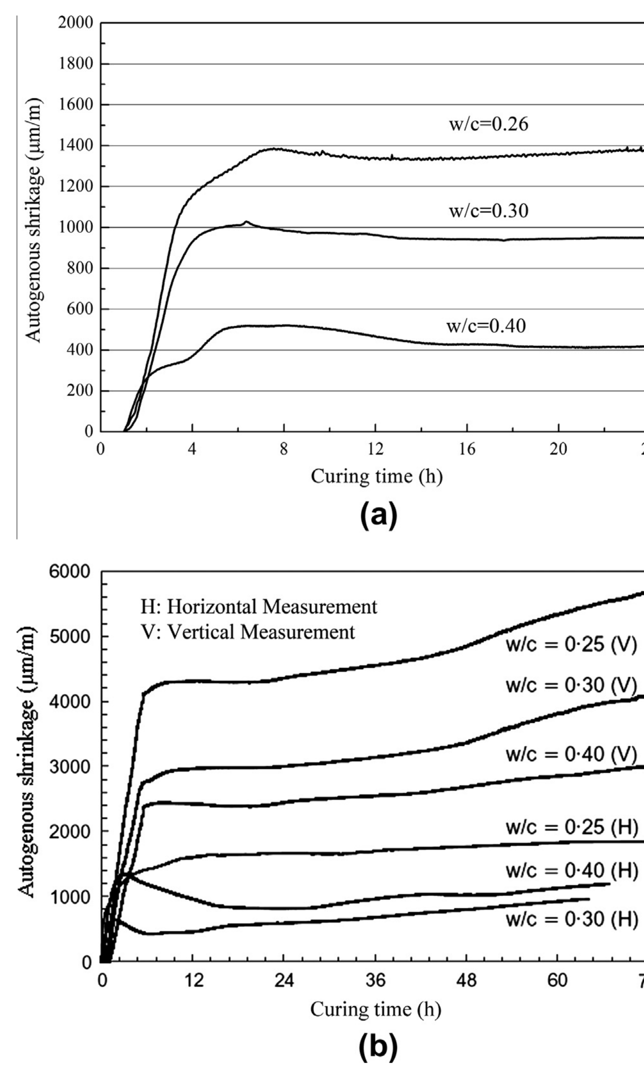

Fig. 8. The influence of w/c ratio on the autogenous shrinkage of Portland cement paste. (a) Results given by the present system, and (b) results given by Bouasker et al. [16].

autogenous shrinkage of cement paste (type I Portland cement) with the w/c ratio of 0.25 after 24 h reached about 1400 μm/m, being significantly higher than that of cement pastes with the w/c ratios of 0.30 (about 900 μm/m) and 0.40 (about 600 μm/m), as shown in Fig. 8(b). Holt [7] illustrated that chemical shrinkage was constant for equivalent cement contents at the early age. The chemical shrinkage of the cement paste with a lower w/c ratio was increased, because it had a higher cement contents. Consequently, the autogenous shrinkage of cement paste with a lower w/c ratio was increased at the early age.

4. Conclusions

Main conclusions that can be drawn from the present study are as follows:

(1) Test methods for the measurement of autogenous shrinkage of cement paste were reviewed, and it is found that few methods are suitable to measure the autogenous shrinkage of cement paste at very early age.
(2) A non-contact system for autogenous shrinkage measurement of cement paste at very early age was designed and established by using non-contact LDVT sensors and LDPE corrugated tubes.
(3) The transformation ratio of volume deformation to length change of the designed non-contact system can be as high as 0.97, which is much higher compared to the contact system.
(4) The designed non-contact system can measure the autogenous shrinkage of cement paste immediately after casting. A typical standard deviation of results before and after setting was approximate 50 μm/m and 10 μm/m, respectively. Compared to the contact system, this designed non-contact system exhibits an excellent repeatability.

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