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A review on testing methods for autogenous shrinkage measurement of cement-based materials

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A review on testing methods for autogenous shrinkage measurement of cement-based materials

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Autogenous shrinkage is an important factor for cracking of cement-based materials at early ages. This review summarizes different definitions and testing methods for autogenous shrinkage in literature. Autogenous shrinkage should be defined as the sum of chemical shrinkage and self-desiccation shrinkage. All testing methods in the literature can be divided into two categories: direct and indirect testing methods. Direct testing methods usually measure volumetric or linear dimensional changes. Indirect methods usually measure porosity or change in relative humidity of cement-based materials, which can reflect the change in autogenous shrinkage. Indirect methods need to establish correlations between autogenous shrinkage and porosity or relative humidity.

Keywords: cement-based materials; autogenous shrinkage; chemical shrinkage; self-desiccation shrinkage; testing methods

1. Introduction

Autogenous shrinkage was first described by Lynam [1] in the 1930s, which later became known as "autogenous volume change".[2,3–5] However, it did not catch much attention until recently, when early cracking often happened in high-performance concrete mixtures.[1–5] In the past few decades, several authors have proposed different definitions for autogenous shrinkage. The significant ones are listed in (Table 1). They can be classified into three main categories: (1) autogenous shrinkage refers to the chemical shrinkage or Le Chatelier contraction derived by the difference in density between hydration products and reactants [3,4,6]; (2) autogenous shrinkage basically corresponds to the decrease in the solution surface tension and leads to the change of capillary pressure, namely self-desiccation [5,7–9]; and (3) autogenous shrinkage is mainly associated with chemical shrinkage and self-desiccation.[1,10,11] In spite of the confusions among those definitions, it is widely accepted that autogenous shrinkage should be independent of variation in temperature, moisture content, external loading, and restraints.

Cement-based materials go through three different stages at early ages after mixing with water: plastic, setting, and hardening.[12] In the plastic stage, all pores within cement paste are filled with

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Table 1. Definitions of autogenous shrinkage in the literature.

Author	Definition	Year	References
Lynam	Shrinkage that is not due to thermal causes, stresses caused by external loads or restraints, or the loss of moisture to the environment	1934	[1]
Davis	Change in volume produced by internal physical and chemical reactions	1940	[3]
ACI 116R	Change in volume produced by continued hydration of cement, exclusive of effects of applied load and change in either thermal condition or moisture content	1985	[4]
Tazawa	A phenomenon in which cementitious materials shrink at a constant temperature without any change in weight	1995	[5]
Japan Concrete Institute	Microstructure volume changes because of hydration of cement after internal setting	1999	[6]
RILEM TC	External volume decreases because of hydration of cement	2002	[10]

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water. The autogenous shrinkage is mainly composed of chemical shrinkage resulting from the hydration of cement.[13,14] It has little effect on cracking and is harmless. Moreover, many researchers do not include this stage during their autogenous shrinkage measurement. As soon as a rigid structure develops, capillary pressure starts to develop and menisci is formed in capillary pores, which causes pore pressure and volume change.[15,16] At this stage, some water in capillary pores within cement paste is consumed, and shrinkage due to self-desiccation becomes obvious. Once the paste starts to harden, change in pore pressure and volume of cement paste become significant. Then the paste may generate internal cracking because it has the limited constraining ability to deformation. Many factors, such as water-to-cement (W/C) ratio and the addition of supplementary cementitious materials or chemical admixture, have a great influence on autogenous shrinkage at early ages.[12,17,18] Davis [3] noted that autogenous shrinkage of ordinary Portland cement concrete was about 40 μm/m, much smaller than drying shrinkage which ranges from 400 to 800 μm/m. Aıtcin et al. [19] found that when W/C was lower than 0.42, autogenous shrinkage increased rapidly.

Different definitions for autogenous shrinkage also cause confusions in measurement. Some definitions define autogenous shrinkage as the shrinkage starting from initial setting.[15] However, the times for initial setting of different concrete mixtures are different and difficult to determine accurately. On the other hand, the shrinkage after initial setting also includes those resulting from the hydration of cement and self-desiccation. Thus, it causes difficulty in proper determination and measurement of autogenous shrinkage. Many researches began autogenous shrinkage measurement one day after casting.[14] Actually, autogenous shrinkage occurs as soon as water and cement are mixed.[15] Thus, autogenous shrinkage should be defined as the total shrinkage from both hydration of cement and self-desiccation starting from mixing, which is more clear and easier for measurement; but, only those happening after setting can cause cracking.

Many testing methods have been proposed or standardized for autogenous shrinkage measurement. They can be

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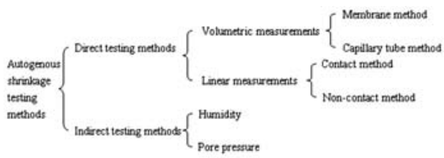

Figure 1. Classification of testing methods for autogenous shrinkage measurement.

classified into direct and indirect methods, as shown in (Figure 1). In this paper, all these methods are briefly described and discussed for their advantages and disadvantages. Finally, the needs for further development are proposed.

2. Direct testing methods

2.1. Volumetric measurement

Volumetric measurement measures volume changes of cement-based materials at constant temperature. They can be classified into membrane and capillary tube methods, as described below.

2.1.1. Membrane method

Membrane method (or buoyancy method) was first used by Yates [20] in the 1940s based on the change in buoyancy of the specimen in a liquid. After filling with fresh cement paste, a plastic membrane (or rubber) bag is sealed and immersed into a liquid immediately. Autogenous shrinkage is assessed by recording the weight change of the plastic membrane over time.

Water bleeding of the cement paste and change in environment temperature can have an obvious effect on the accuracy of the measurement in this method, especially when W/C is high.[14] Reabsorption of bleeding water on the surface of the specimen and change in temperature due to hydration of cement cause invalidate results. In 1994, Justnes et al. [13] developed a testing apparatus coupled with temperature controller based on Yates' method, as shown in (Figure 2). Some researchers placed the film bag

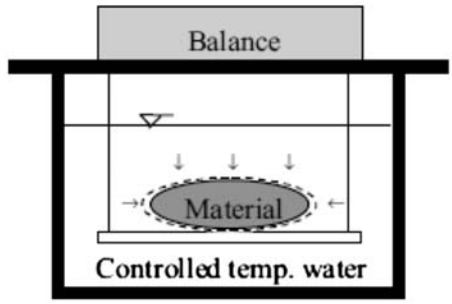

Figure 2. Measurement of autogenous shrinkage using rubber bag [52].

filled with cement paste on rotation to avoid bleeding.[21,22] In this way, bleeding water can be reduced, but cannot be completely eliminated. To improve the repeatability and accuracy of the measurements, Loukili et al. [21] used a computer to control the temperature of water bath and to monitor the change in weight. Later, further attempts were made to avoid water level change and thermal gradient of water bath.[22,23]

Buoyancy method is easy to perform in the lab. However, there are two potential problems with it: (1) the frail rubber bag is only suitable for paste and mortar, not for concrete; and (2) the influences of heat release and capillary tension caused by the hydration of cement-based materials are ignored.

2.1.2. Capillary tube method

Capillary tube method is normally used for measuring Le Chatelier contraction. A setup developed by Tazawa [5] is showed in (Figure 3). Typically, fresh cement paste is placed in tubes of different sizes. Oil or water is immediately added onto the surface of the paste in the tube. Then, autogenous shrinkage is recorded with a measuring pipette inserted on top of the paste. It is thought that pure chemical shrinkage is measured when water is put on the top surface of cement paste, since additional water in the sample can be

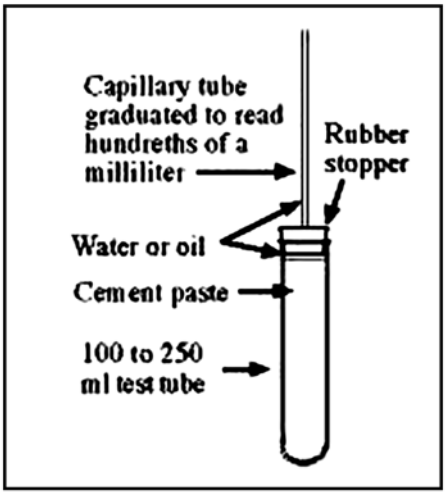

Figure 3. Measurement of chemical shrinkage of cement paste [53].

supplied for the hydration of cement.[5,7] However, the external water cannot reach the middle and bottom portions of the sealed paste. Self-desiccation develops from those portions. When oil is used, recorded shrinkage includes both chemical and autogenous shrinkage. The portion of chemical shrinkage from use of oil is smaller when compared with the use of water.

Although capillary tube method is simple and easy to perform, measured results may be very variable due to thermal effects, especially when the sample is large. It was found that the thickness of the specimen with low W/C had a great effect on measured results.[24,25]

Volumetric measurement can start data recording immediately after casting, but their limitations are obvious.[26] Cement paste may be too weak to overcome the friction of tired rubber bag at early ages.[27] Bleeding water and entrapped air also obstruct significantly, if bleeding water is sucked back into the cement paste during the hydration of cement.[28] Ultimate volume reduction in the immersed cement paste may be erroneously measured. Temperature control of cement paste is still yet to be successfully solved, which results in unrepeatable results.

2.2. Linear measurement

Linear measurement measures the length change of cement paste, mortar, or concrete specimens cast in a rigid steel mold and recorded by sensors at one end or both ends of the specimens. They can be classified into contact and noncontact methods according to the sensors used or horizontal and vertical methods, according to the displacement of specimens. The horizontal method is more commonly used because it can reduce the influence of gravity during the measurement. The specimens used can be either prisms or cylinders. There are standard testing methods in different countries.[8,9,29,30]

2.2.1. Contact method

Contact method uses gauges or sensors embedded in or in direct contact with specimens for shrinkage measurement. The Japan Concrete Institute [6] used horizontal prisms of 100 × 100 × 400 mm and two dial gauges connected with bolt as data loggers at both ends. It measures the shrinkage of concrete with rigid mold accurately, especially for large specimens. The specimens need to be removed and sealed by aluminum foil 24 h after casting. To address the problem of thermal related deformations, the JCI simply used a thermal coefficient for correction, say 10 μ/°C for concrete. However, it was not reliable. Bjontegaard [31] introduced water circulation through tubes to control the temperature of the specimens. To determine the starting time point of autogenous shrinkage, someone also tried to measure the setting time simultaneously on a separate sample.[32]

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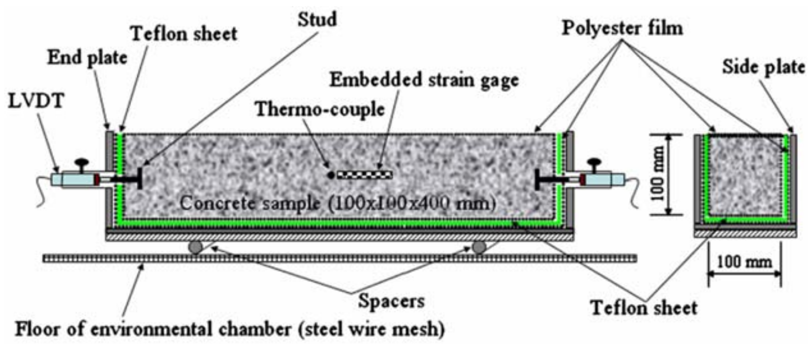

Figure 4. Setup with LVDTs for early autogenous shrinkage measurement [33].

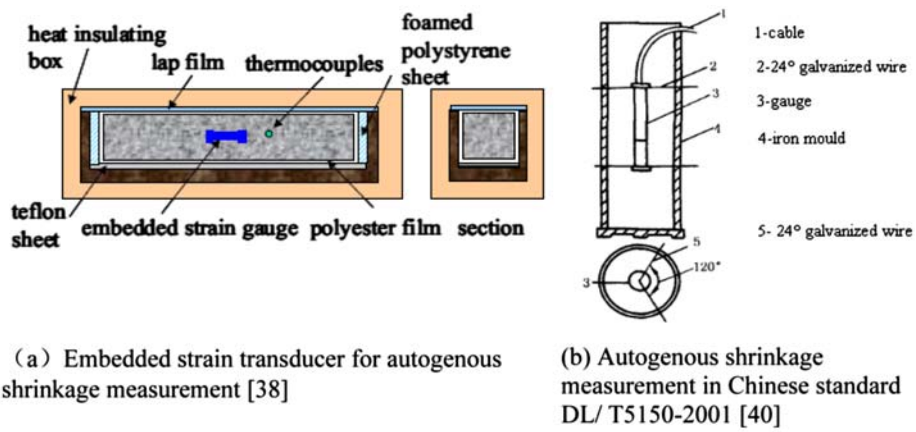

Figure 5. Embedded strain transducer for autogenous shrinkage measurement.

Later, some researchers used linear variable differential transformer sensors instead of dial gauges [33] and got more accurate results, as shown in (Figure 4). Recently, inductance frequency-modulation type microdisplacement sensors were used to convert shrinkage into electric signal with measurement range of up to 2000 μm.[32] In this method, sensors need to be chosen based on actual measurement range to obtain more accurate measurements.

Yang [34,35] and Chinese standard DL/T5150-2001 [29] measured shrinkage of cement-based materials by embedding sensor and thermocouple in horizontal specimen of $100 \times 100 \times 400$ mm and vertical specimen of $40 \times 40 \times 100$ mm, as shown in (Figure 5). To reduce the influence of hydration heat and friction between concrete and steel plate, Yang placed 1 mm-thick Teflon film in the template and 2 mm-thick foamed polystyrene between polyester film and plate. The most crucial problem associated with this method is to place sensor and thermocouple at the right position in the specimen, because fresh specimen may not be strong enough to support the embedded sensors. Moreover, this measurement can be expensive because the embedded thermocouple and strain gage cannot be reused.

In 2007, Allan [35] proposed a testing method with optical fiber Bragg grating (FBG) sensor connected to the

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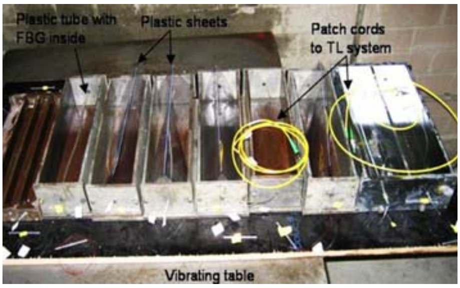

Figure 6. FBG sensors for autogenous shrinkage measurement [41].

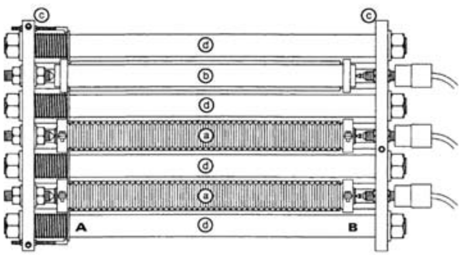

Figure 7. Bellows for autogenous shrinkage measurement [7].

tunable laser system used for shrinkage and temperature monitoring, as shown in Figure 6. Because of the small size, FBG sensors are noninvasive to the specimens. Moreover, the measurement can start at the very beginning stage of cement-based materials after being cast.

In general, linear measurement is a more realistic representation of material behavior and the actual field construction because thermal dilation, bleeding, setting time, and other factors are all accounted. It is also possible to separate the deformation resulting from thermal changes associated with cement hydration to obtain autogenous shrinkage. However, settled concrete can exert vertical force on a steel mold located on the concrete surface, which prevents the free deformation in this direction. Thus, this method is only suitable for measurement after cement-based material has hardened.

In 1999, Jensen and Hansen [7,36] developed corrugated tube method referring to volumetric measurement and linear measurement method, as shown in Figure 7. They used a mold with length-to-diameter ratio of about 400:30 mm. Specimen is placed in ethylene glycol solution at 20 ± 0.1 °C immediately after fresh mixture is injected into the bellows. Therefore, the flow ability of the pastes needs to be guaranteed in order to carry out properly. The measurement started about 30 min after water addition. Because the deformation in axial direction far overweighs that that in radial direction of the bellow, the volume deformation is the same as the length deformation.[7,37] Moreover, the corrugated tubes or bellows adopted can minimize the restraint on the specimens. The results from this method were stable and repeatable, especially for cement-based materials with certain hardness. Recently, Tian Qian [37] did a systematic study on factors affecting autogenous shrinkage measurement using bellow method. She found that the length of tubes and internal air bulbs had little effect on the measurements; but, the stiffness of tubes had a significant effect on the measurements at early stages. Moreover, the most suitable size and stiffness for bellows were proposed through large number of experiments.

Some researchers used vertical specimens in their measurements with dial gauge or sensors placed vertically. Among them, the most widely used one was that developed by Nawa [38] and his colleagues. Free deformation of the specimen can be ensured by embedded perpendicular thermocouple and sensors. However, the weight of sample and the starting time of measurement can have some effects on the measured results.

2.2.2. *Noncontact methods*

It is well known that the use of contact sensors may damage cement-based materials at early stages and produce relative

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displacement when measuring heads are inserted. Noncontact methods such as the laser displacement sensor and the eddy current sensor methods are the best options to solve these problems. These sensors are suitable for bad industrial environments, since they are not sensitive to oil pollution, dust, humidity, and interference of magnetic field. Moreover, the results have good accuracy and stability.[39] Park [40] developed a method using laser displacement sensors with specimen of $40 \times 40 \times 160\,\text{mm}$, as shown in (Figure 8). The results from this method could have certain deviation, if the specimens are not strong enough.

In 2005, Tian Qian and Sun Wei [41] designed an apparatus for the measurement of autogenous shrinkage of concrete at early stages. The measurement includes two stages: (1) a horizontal noncontact sensor was used for autogenous shrinkage during the first 24 h; (2) a vertical dial indicator was used after one day. This method was effective not only to avoid mold constraint, vibration, and interference, but also to collect and analyz data automatically. However, the molds and sensors need to be changed.

In summary, linear measurement can reflect the actual behavior of materials compared with volumetric measurement. The measured results are more stable since studs are fixed on specimens during the measurement. On the other hand, volumetric measurement can start as soon as cement paste is poured into a membrane or glass container. Among volumetric measurement, buoyancy method is more commonly used even though the results are difficult to control. The corrugated tube method proposed by Jensen and Hansen can turn volume change into length change and begin measurement immediately after casting. Thus, it is the most reasonable method for measurement of autogenous shrinkage so far.

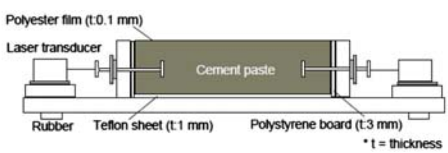

Figure 8. Setup for horizontal autogenous shrinkage measurement.

3. Indirect testing methods

During the hydration of cement-based materials, two types of pores are formed: gel and capillary pores. Among them, the size of gel pore is in nanometer range when internal relative humidity is above 80%.[42–44] It means that when the relative humidity is between 80 and 100%, the surface tension of nanometer-sized pores does not work, because water loss is mainly from capillary pores during drying.[54,55] Though it is generally accepted that occurrence of autogenous shrinkage is closely related to relative humidity and pore pressure of specimens, there are limited researches on the relationships between them.

Previous studies [45,46,56] found that there was remarkably a linear relationship between relative humidity hs and autogenous shrinkage εs, as expressed by Equation (1):

$$\varepsilon_s(h_s) = mh_s + n \qquad (1)$$

where, m and n are constants depending on W/C ratio, volume, and content of the mineral admixture.

Barcelo [47] designed a method to measure the change in relative humidity to estimate autogenous shrinkage, as shown in Figure 9. A specimen of

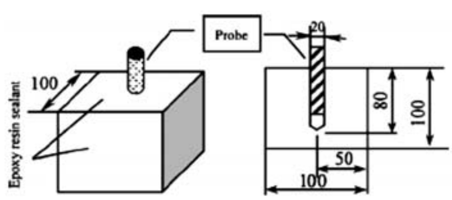

Figure 9. Setup for measurement of change in relative humidity [44].

$100 \times 100 \times 100\,\text{mm}$ was sealed with 2 mm layer of wax, above which the relative humidity probe was embedded.

According to Kelvin's equation, relationship between relative humidity and meniscus radius of capillary pores can be expressed by Equation (2) [47]:

$$\ln(RH) = \frac{-2\gamma V_{\text{m}}}{rRT} \tag{2}$$

where RH is relative humidity, γ is the surface tension of pore solution (N/m), r is meniscus radius of capillary pores (m), R is gas constant (8.314 J/ (mol. K), and T is temperature (K). Equation (2) shows that the smaller the concrete pore size, the lower the internal relative humidity.

Autogenous shrinkage and porosity of cement-based material are closely related. MacKenzie [48] and Bentz et al. [49,50] proposed an equation to estimate shrinkage produced by capillary tension of cement-based material as expressed by Equation (3):

$$\varepsilon = \frac{S\sigma_{\text{cap}}}{3}\left(\frac{1}{K} - \frac{1}{K_{\text{s}}}\right) \tag{3}$$

where ε is linear strain or shrinkage, S is the degree of saturation; K is bulk modulus of elasticity of porous material (Pa), and K_{s} is the modulus of elasticity of solid frame of porous materials (Pa). The equation is applicable to fully saturated linear elastic material. Capillary tension can be calculated by Equation (4):

$$\sigma_{\text{cap}} = \frac{2\gamma}{r} \tag{4}$$

It was Wittmann [51] who first attempted to establish the relationship between autogenous shrinkage and porosity. Later on, other people did some further studies and proposed a quantitative relationship as shown by Equation (5) [1,4]:

$$\frac{\partial \varepsilon_{\text{a}}}{\partial t} = \lambda \frac{\partial \sigma}{\partial t} \tag{5}$$

where, $\partial\varepsilon_{\text{a}}$ is the increment of autogenous shrinkage, $\partial\sigma$ is the change of surface tension, and λ is scale factor.

Generally speaking, estimated autogenous shrinkage from indirect methods using this model is consistent with the actual measured results. Early chemical shrinkage has to be measured by other methods since relative humidity, pore pressure, and porosity are mainly related to drying shrinkage.

4. Conclusion

According to various definitions and measurement methods for autogenous shrinkage in literature, autogenous shrinkage of cement-based material should refer to macroscopic volume change at constant temperature with no moisture exchange with the external environment. It includes both chemical and self-desiccation shrinkage.

Testing methods for autogenous shrinkage measurement can be divided into direct and indirect methods. Direct methods include volumetric and linear measurement. Among volumetric methods, buoyancy method is the earliest and the most common used one. However, it is mainly suitable for small cement paste or mortar samples. Capillary tube method measures both chemical and autogenous drying shrinkage.

Linear measurements can be divided into contact and noncontact ones. The former usually have high accuracy when sensors are embedded in samples. At the same time, temperature should be monitored for calibration. Moreover, the numerical error is smaller when noncontact method is used, since there is no constraint to specimens. Corrugated tube method can start measurement immediately after casting. It is less dependent on

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the environment and the best autogenous shrinkage measurement method so far.

Autogenous shrinkage can be estimated by indirect measurement of porosity and change in relative humidity. Results from indirect methods are consistent with those from direct ones. However, chemical shrinkage at early ages cannot be measured by indirect measurements.

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