



Experimental investigation on the autogenous shrinkage of steam cured ultra-high performance concrete

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HIGHLIGHTS

- Key factors that affect autogenous shrinkage of UHPC were steel fiber, silica fume and aggregate.
- The autogenous shrinkage development was divided into 3 stages.
- The ettringite formation is responsible for the slight expansion after steam curing.

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ABSTRACT

The ultra-high performance concrete (UHPC) has the potential to be widely used for its excellent mechanical properties and durability. However, characteristics such as high superfine particles content, free of coarse aggregate and extremely low water to binder ratio lead to high autogenous shrinkage in UHPC that could even cause cracking. In this work, the mixture parameters on the autogenous shrinkage behavior of steam cured UHPC were investigated. High performance liquid chromatography was employed to determine the sulfate concentration at different ages. The development of microstructure and hydration products were investigated by a range of analytical techniques such as X-ray diffraction analysis, scanning electron microscopy and nuclear magnetic resonance spectroscopy etc. The results showed that key factors that affected autogenous shrinkage of UHPC were steel fiber, silica fume and aggregate to cement ratio. The autogenous shrinkage mainly occurred during steam curing, and a slight expansion behavior was observed after steam curing. The microstructure observation showed that the hydration was significantly promoted by steam curing and $\text{Ca}(\text{OH})_2$ can be rarely observed. A very dense microstructure and closely bonded interface between aggregate and the matrix was observed. The decrease of sulfate concentration and appearance of a weak shoulder at about 13 ppm in the ^{27}Al MAS NMR spectra of sample at 60 d indicated that ettringite formation was responsible for slight expansion after steam curing.

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1. Introduction

As a kind of advanced cement based material developed at Bouygues in 1990s [1], Apart from the mechanical properties, the UHPC possesses high fracture capacity, good permeability resistance [2], excellent freezing and erosion resistance [3], and high fire endurance temperature [4–6]. Therefore, it is expected that the UHPC will be widely applied in fields of bridge, nuclear power station, super

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high-rise building and military protection construction, etc. However, due to the present of high fraction of superfine particles, low water to binder ratio and free of coarse aggregate of UHPC [7], the risk of high shrinkage cannot be ruled out. Generally, such high shrinkage will result in residual tension that may cause the formation of cracks and further impair the durability of UHPC [8,9].

There are many kinds of shrinkage that can influence the crack behavior of concrete, such as drying shrinkage, autogenous shrinkage, chemical shrinkage, carbon shrinkage and plastic shrinkage. Among them, the autogenous shrinkage is a kind of early shrinkage caused by the loss of water from capillary pores due to hydration. For normal concrete, the autogenous shrinkage is usually less than 100×10^{-6} m/m at the age of 1 month, which is much lower than

that of drying shrinkage [10]. However, the autogenous shrinkage develops rapidly in concrete with low water to binder ratio, and eventually becomes the dominant role of total shrinkage [11–13]. A high autogenous shrinkage value of $700 \times 10^{-6} \text{ m}\cdot\text{m}^{-1}$ for concrete with water to cement ratio of 0.17 was reported [14]. In consequence, for UHPC prepared with a very low water to cement ratio and high silica fume content, the total shrinkage is certainly attributed to the autogenous shrinkage [15]. Meanwhile, for steam cured UHPC, the microstructure and hydration products characteristic are different from that cured at normal temperature [16], which may lead to variation of the shrinkage behavior and mechanical behavior [17]. In addition, the high temperature not only accelerates the hydration process but also promotes the increase of autogenous shrinkage [18,19]. Therefore, it is of great significance to investigate the autogenous shrinkage behavior of steam cured UHPC.

Generally, the autogenous shrinkage occurs when self-desiccation leads to the emptying of pores inside the bulk cementitious material, and the self-desiccation is attributed to withdrawal of water from the capillary pores due to hydration [20]. Various studies were done to investigate the autogenous shrinkage behavior of normal concrete or cement paste [15,21–26]. For UHPC, the autogenous shrinkage behavior was also reported in some previous research. For example, Ahmad et al. investigated the effects of parameters including the water to binder ratio, cement content and age on the autogenous shrinkage of UHPC at 20 °C [27]; And Ma and co-workers compared the autogenous shrinkage of concrete containing coarse aggregates to that of UHPC at room temperature, a decrease rate of 60% was observed after adding coarse aggregate [28]. Moreover, approaches to reduce this autogenous shrinkage were also proposed. Materials including expansive additive, shrinkage reducing admixtures, superabsorbent polymers were utilized to reduce the autogenous shrinkage of UHPC prepared at normal temperature [29,30]. However, there are few studies that focus on the autogenous shrinkage behavior of steam cured UHPC, and most of current research are mainly focused on the autogenous shrinkage under normal temperature. Also, the effects of different mixture proportions on its autogenous shrinkage behavior of steam cured UHPC are still unclear. If the relationships between the mixture parameters and autogenous shrinkage behavior of steam cured UHPC are quantitatively correlated, the autogenous shrinkage of steam cured UHPC could be controlled in a reasonable value by optimizing the mixture proportion, which will further decrease the risk of cracking.

Consequently, based on these premises, the objective of this research is to investigate effects of water to binder ratio, aggregate content, aggregate size, silica fume content and steel fiber dosage on the autogenous shrinkage behavior of steam cured (at 90 °C) UHPC. And after the temperature dropped back to 20 °C, the autogenous shrinkage was also monitored to 60 days. Furthermore, the mechanical properties of UHPC were measured at different ages. Meanwhile, the high performance liquid chromatography (HPLC) was employed to determine the sulfate ion concentration of UHPC specimens at different ages. Additionally, several other techniques including scanning electron microscopy (SEM), X-ray diffraction (XRD), and ^{27}Al nuclear magnetic resonance spectroscopy (^{27}Al MAS NMR) were applied to characterize the development of microstructure and hydration products of steam cured UHPC.

2. Materials and methods

2.1. Materials

The Portland cement produced by Huaxin cement Co., LTD was used to prepare the UHPC and pastes. The silica fume produced by WISCO group was used. The chemical compositions of the Portland

cement and silica fume are listed in the Table 1 and their technical properties are listed in the Table 2. Quartz sand with a diameter ranging from 0.125 to 2 mm were used as aggregate. Steel fiber (length = 13 mm, diameter = 0.22 mm), superplasticizer and quartz powder were used. Clean tap water was used for all mixtures.

The recipes of UHPC we used is listed in Table 3. The effect of water to binder ratio, aggregate content (represent as aggregate to cement ratio), maximum size of aggregate, silica fume content and steel fiber dosage on the autogenous shrinkage were considered. The water to binder ratios of 0.16, 0.18, 0.20, 0.22 and 0.24, the quartz sand with the maximum diameter of 0.212, 0.425, 0.85 and 2 mm (The gradation of quartz sand of different maximum sizes is shown in Table 5), the replacement of cement by 10%, 20% and 30% silica fume, and the steel fiber with volume fractions varied from 0% to 4% were chosen. Also, the aggregate (quartz sand) to cement ratios of 0.7, 0.9, 1.1, 1.3 and 1.5 were used. UHPC pastes without quartz sand was prepared according to UH0 for XRD analysis and ^{27}Al NMR testing. The specimens prepared according to the mixture proportion in Table 3 were used to apply scanning electron microscopy and high performance liquid chromatography test.

The mix procedure was chosen based on the literature [31]. The dry powders, including cement, silica fume and quartz sand were first mixed for 3 min at a high speed. Then water and superplasticizer were added and mixed for approximately 5 min at a low speed. After mixing, the mixtures were poured into the molds and consolidated using a vibrating table. The UHPC mixtures were placed in molds sizing of 40 mm × 4 mm × 160 mm and the UHPC pastes mixtures were placed in molds sizing of 40 mm × 4 mm × 40 mm. After casting, all specimens were stored in a room (20 °C and 100% RH) for 24 h prior to demolding. In addition, the specimens were sealed by several layers of low density polyethylene (LDPE) and a layer of polypropylene after demolding at 24 h. Then, they were placed in a steam-curing chamber to cure for 48 h at 90 °C. The detailed curing process is illustrated in Fig. 1. After steam curing, the specimens were cured at 20 °C. Also, specimens cured with standard condition (20 °C and 100% RH) are prepared according to the mixture proportion shown in Table 3.

2.2. Methods

2.2.1. Autogenous shrinkage

Contact shrinkage deformation of concrete tester was used to determine the autogenous shrinkage of UHPC. The size of specimen is 40 mm × 4 mm × 160 mm. The shrinkage measuring apparatus used to determine the autogenous shrinkage of UHPC is the concrete length comparator, which consists of a semi closed metal box attached to a metal base and a reference bar, as shown in Fig. 2 (a). The comparator is designed to measure the length of concrete samples quickly and accurately, then the autogenous shrinkage could be calculated based on these measurements. The first autogenous shrinkage value was measured immediately after demolding at 24 h, and the second one was measured at 3 d after cooling down to 20 °C. Other values were obtained at 20 °C. These measurement were continued until the 60th day. In addition, all specimens were sealed by several layers of low density polyethylene (LDPE) and a layer of polypropylene when the steam curing and shrinkage test were conducted. The autogenous shrinkage was tested under sealed condition.

Non-contact shrinkage deformation of concrete tester, model CABR-NES, was used to determine the development of autogenous shrinkage of UHPC. Fig. 2(b) shows the details of the test setup. The shrinkage or expansion could be monitored by two steel bars in the concrete. The fresh concrete mixture was casted into the mold and covered with a polyethylene film to prevent possible water evaporation.

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