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Nano-mechanical behavior of a green ultra-high performance concrete

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HIGHLIGHTS

- The UHPC contained high volumes of fly ash and used river sand as aggregate.
- The nano-mechanical behavior of the UHPC was investigated by nanoindentation.
- The hydration products of the UHPC are dominated by high-stiffness hydrate phases.
- The mechanical bond at the interfacial zone is strong and efficient.
- High-stiffness phases such as mullite and hematite in fly ash were detected.

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ABSTRACT

As a revolutionary construction material, ultra-high performance concrete (UHPC) has been extensively studied in the last two decades and one research interest has been focused on preparing UHPC with low-priced raw materials or industrial by-products. In this study, a green UHPC which used high volumes of fly ash and river sand as part of raw materials was prepared, and the objective is to investigate the nano-mechanical behavior of the UHPC using nanoindentation. The results show that the hydration products, which account for about half of the paste by volume, are mainly high-stiffness hydrate phases, and significant quantities of unreacted cement and fly ash have higher mechanical properties than the hydration products and can function as micro-aggregates to strengthen the UHPC paste. Moreover, the mechanical properties of the paste near aggregate or fiber surfaces are similar to those of the bulk paste, which indicates that the UHPC has a strong and efficient bond at the interfacial zone. The experimental findings at the nano-scale could help to understand the macro-performance of the green UHPC.

1. Introduction

Among the recent advancements in concrete technology, one remarkable achievement is the development and increasing use of ultra-high performance concrete (UHPC). UHPC is a highly compact, dense material that exhibits attractive flowability, excellent mechanical properties and exceptional durability. UHPC was first developed in 1995 by Richard and Cheyrezy [1] and in the past few years it has been applied to coupling beams in high-rise buildings, precast members, infrastructure rehabilitations, blast resistant structures and special facilities like nuclear waste storage containers. It is regarded as an innovative and promising material for the future, yet the high initial cost affects its broader application. In this regard, the use of industrial by-products such as fly ash and slag to partially substitute for cement in UHPC production have been extensively studied, and the replacement of costly quartz sand by natural sand or industrial wastes like glass cullet and iron ore tailings have also been reported [2–6]. The incorporation of these materials into UHPC not only fulfills the economic requirement, but also brings other advantages such as lowering the shrinkage and improving the durability of the material. More importantly, the substitution contributes to the recycling of industrial waste and the reduction of cement consumption, which reduces CO_2 emission and makes the material environmentally friendly.

The superior performance of UHPC originates from its engineered microstructure, which is obtained by eliminating coarse aggregate to improve homogeneity, maximizing the packing density with an optimized gradation of granular constituents and enhancing the ductility with high-strength fibers [1]. The microstructure of UHPC has been characterized by multiple experimental techniques including secondary electron microscopy (SEM), X-ray diffraction (XRD), mercury intrusion porosimetry and thermogravimetric analysis [7–9]. While the experimental results reveal that UHPC has a much denser microstructure and contains much less calcium hydroxide (CH) crystals as compared

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with ordinary concrete, the underlying mechanisms that govern the macro-performance are still not very clear. The main binding phase, calcium silicate hydrates (C–S–H), is reported to be a porous material with two different packing densities, known as low density (LD) C–S–H and high density (HD) C–S–H [10,11]. Their differences in microstructure could significantly affect the material performance, yet their properties cannot be studied in depth by the aforementioned conventional methods. Furthermore, the very low water-to-binder ratio (w/b) of UHPC implies that a large quantity of cementitious materials remain unreacted after curing, which raises the question of what role the unreacted particles play in the material.

Recent applications of nanoindentation in cement-based materials can help unveil the mystery of the materials at the micro- or nano-scale. Nanoindentation has been proved to be a reliable tool to determine the intrinsic mechanical properties of various phases in cement-based materials [12–15]. In this respect, the mechanical properties of cement grains and hydration products have been studied intensively but few research works have been reported for mineral admixtures like fly ash. Another noticeable application of nanoindentation is to quantitatively characterize the interfacial transition zone (ITZ), whose thickness is reported to be several tens of micrometers [15–18]. Compared with microindentation, the relatively small interaction volume during penetration makes it more appropriate and advantageous to get an accurate profile of the mechanical properties at the interfacial zone. By virtue of the nanoindentation technique, this paper investigates the nano-mechanical behavior of a green UHPC formulae which contains high volumes of fly ash and uses natural river sand as fine aggregate. The experimental results are considered to help understand the macro-performance of the UHPC material.

2. Materials and sample preparation

2.1. Materials

The UHPC studied here is applied to produce cover slabs which are ancillary elements of the rapidly-growing high-speed railway in China. The cover slabs are used to cover the electric cable trenches and provide a pathway for pedestrian and track maintenance. Fig. 1a shows a picture of the cover slabs along the rail, and Fig. 1b shows the dimensions of a typical slab. The slabs are designed to have adequate mechanical properties to bear the applied loads and a high durability to provide a long service life. The required properties for the UHPC are summarized in Table 1. Conventional slabs are made of ordinary reinforced concrete which frequently deteriorates due to a poor durability. In contrast, though the initial cost of using UHPC slabs is higher, the long service life and low maintenance cost could contribute to a lower life cycle cost. Moreover, the significant material savings (dead weight reduced by 40%) could possibly lead to a lower embodied energy.

The mix design of the UHPC is given in Table 2. Ordinary Portland cement, type F fly ash and silica fume were used as the cementitious materials, and their physical properties and chemical composition can be found at [6]. In particular, the mineralogical composition of the fly ash was examined by XRD and is given in Fig. 2. The XRD pattern displays a series of sharp diffraction peaks, identified as mullite, quartz, and minor quantities of hematite and lime. In addition to those crystalline phases, a pronounced broad hump caused by diffuse scattering of X-rays can be observed in the background between 16° and 35° 2θ, which indicates the presence of amorphous vitreous materials. Natural river sand with an apparent density of 2.63 g/cm^3 and a maximum particle size of 5 mm was used as fine aggregate. The steel fiber was brass-coated with a length of 13 mm and an aspect ratio of 65. The mixing water was potable tap water, and a polycarboxylate-based superplasticizer was used. Compared with reactive powder concrete [1], the UHPC used a large amount of fly ash to partially replace cement, and employed natural river sand as substitute for costly quartz sand. The cost of the material is lowered and the energy consumption is reduced. Therefore, the UHPC material promotes green benefits with less CO_2 emission.

2.2. Sample preparation

In this study, both the UHPC paste and UHPC were prepared and tested to investigate the nano-mechanical behavior of the cementitious phases and the nano-mechanical behavior of the interfacial zone, respectively. The rationale behind using the paste sample was to avoid possible disturbance by indentation response from the aggregate. Moreover, to ensure that small sand particles slightly beneath the surface would not affect the results at the interfacial zone, sand particles smaller than 0.3 mm was removed by a standard sand sieve.

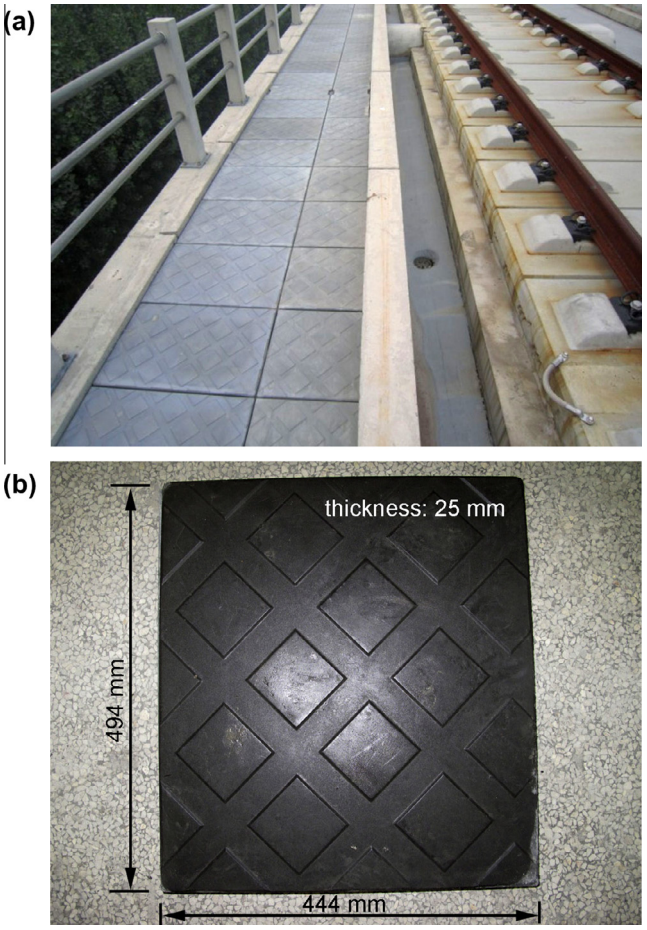

Fig. 1. Sidewalk along the high-speed rail (a) and dimensions of a typical cover slab (b).

A mortar mixer with two different revolving speeds (140 rpm and 285 rpm) was used to prepare the material. The dry materials were first mixed at low speed for 2 min, which helped to break apart the agglomerates and homogenize the material. Then water and superplasticizer were gradually added and the fresh material was mixed at the high speed for another 5 min to obtain good flowability. Afterwards, the mixture was cast into a PVC tube with a diameter of 15 mm and a length of 20 mm. For preparing the UHPC specimen, fibers were not added during mixing but were manually added in parallel with the length direction of the tube after casting, which facilitated locating a round cross section of the fiber after trimming the specimen. The specimens were compacted after molding and cured for 1 day at 20 °C and relative humidity of greater than 95%. Then the specimens were demolded and cured in a steam chamber at 80 °C for 2 days. After curing, the specimens were soaked in ethanol for 7 days to arrest hydration. Then the specimens were oven-dried at 50 °C for 2 days to remove the ethanol before being stored in a desiccator for further treatment.

To do a nanoindentation test, proper surface preparation is critical in order to obtain as flat a surface as possible to get repeatable results [22]. In this study, the cylinder specimens were initially sliced using a diamond saw into samples that were about 10 mm thick. Then the samples were vacuum impregnated with a low-viscosity epoxy resin. The low permeability of the samples should have given a low penetration depth of the epoxy. After the epoxy solidified, the samples were first ground by successively finer-grained silicon carbide paper. The exposed air voids on the surface of the samples were checked to ensure that no epoxy was left after the grinding procedure, to verify that the epoxy-penetrated layer was removed. Then the samples were polished by water-based diamond suspensions of four successively finer particle sizes (9 μm, 3 μm, 1 μm and 0.05 μm) on a Buehler TexMet cloth. The polishing time for using each of the first three diamond suspensions lasted 15 min, while the final polishing period was 2 h. After each polishing step, the sample was cleaned ultrasonically in an ethanol bath for 5 min to remove any debris left on the surface. The polished samples were kept in a desiccator until testing.