Scientific paper

Experimental-Computational Investigation of Elastic Modulus of Ultra-High-Rise Pumping Concrete

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Abstract

Based on the ultra-high-rise pumping concrete engineering test with pumping height of 407 m and maximum pumping pressure of 19.3 MPa, the variation of elastic modulus of the pumping concrete was analyzed by experiments and computational method. Firstly, the back scattered electron (BSE) image binarization method was used to study the effect of pumping on the composition and pore distribution of the concrete. The effects of the pumping process on the elastic modulus of the paste and interface transition zone (ITZ) were quantitatively evaluated by x-ray diffraction (XRD), back scattered electron energy dispersive spectroscopy (BSE-EDS) and homogenization method, and verified by the test results of nanoindentation. The results showed that the porosity of the paste after the ultra-high-rise pumping decreased by 7.27%, the hydration degree of the cement increased by 6.51%, and the elastic modulus of the paste increased by 3.1 GPa. For the ITZ of the sand, the porosity decreased by 5.3 μm , the average porosity decreased by 15.78%, and the elastic modulus increased by 7.7 GPa. For the ITZ of the gravel, the thickness decreased by 6.91 μm , the average porosity decreased by 16.34%, and the elastic modulus increased by 10.2 GPa. Therefore, the effect of the ultra-high-rise pumping on the ITZ was significantly larger than that of the paste in the concrete. Afterward, the computational method was proposed as the combination of the homogenization method and the Lu and Torquato model. As the result, the elastic modulus of concrete after ultra-high-rise pumping can be precisely predicted on the micro-meso-macro scale due to the promising agreement between the experimental measurements and the numerical results.

1. Introduction

With the emergence of a large number of ultra-high-rise buildings around the world, the pumping height of concrete keeps creating new records and the technology of the ultra-high-rise pumping concrete has been evolving continuously (Kaplan *et al.* 2005; Tan *et al.* 2011). Ultra-high-rise buildings, as landmark constructions in many countries, have strict requirements on the structure and mechanical properties of the concrete materials at micro and macro levels. Influenced by factors such as high pumping pressure and long pumping flow time, the ultra-high-rise pumping concrete showed different flow

patterns and hardening properties before and after pumping (Ede 1957; Choi *et al.* 2014). Therefore, it is an important research topic to clarify the influence of the pumping action on the structure and compositions of the concrete at micro, meso and macro scales.

Most of the research on the pumping concrete focused on the calculation and numerical simulation of the flowability and rheological properties of the fresh concrete, the composition and thickness of the lubricating layer and the pumping pressure loss (Tan *et al.* 2011). Feys *et al.* (2012, 2014) studied the rheological behavior of self-compacting concrete before and after pumping by pumping circuit test. The results showed that the viscosity of the concrete decreased with the increase of pumping pressure and flow rate. Besides, influenced by the dispersing effect of admixtures on cement particles during the pumping process, the yield strength of the fresh concrete increased whereas the plastic viscosity decreased. Choi *et al.* (2013) compared the composition of the concrete before and after pumping, concluding that the hydration degree of the concrete increased, and the air content decrease. As known, there were many factors affecting the strength and elastic modulus of the hardened concrete, such as the material mix ratio (Choi *et al.* 2014; Zhao *et al.* 2018), curing environment and age (Ortega *et al.* 2012). As pumping is an indispensable method of transportation for concrete engineering, its influence on the elastic modulus of the concrete cannot be ignored (Choi *et al.* 2013). Since pumping affects the pore distribution and hydration degree of the concrete directly, the mechanical properties will vary inevitably

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Table 1 Chemical compositions (weight %) and basic properties of cementitious materials.

Chemical composition	Cement	Fly ash	Ultrafine slag	Silica fume
SiO ₂	23.6	52.3	45.7	92.7
Al ₂ O ₃	4.4	29.7	5.8	1.1
Fe ₂ O ₃	2.3	6.5	0.3	0.7
CaO	67.5	11.2	46.6	0.8
SO ₃	2.03	1.5	1.2	0.0
Ignition loss	4.08	3.7	0.26	4.0
Density g/cm ³	3.10	2.78	2.81	2.20

Table 2 Mixture proportions of pumping concrete (kg/m³).

Water	Cement	Fly ash	Slag	Silica fume	Sands	Gravel	HRWRA
181.5	320	150	100	35	780	890	14.7

(Choi *et al.* 2013). At present, there is no in-depth study on the effect of the ultra-high-rise pumping on the elastic modulus of the concrete.

In this study, the authors creatively proposed an experimental-computational method to investigate the variation characteristics of the elastic modulus of the ultra-high-rise pumping concrete. On the side of the experiments, based on the pumping test in an ultra-high-rise building during the construction process, the influence on the micro-composition and structure of the pumping concrete was determined by the back scattered electron (BSE) image binarization analysis method and the nanoindentation test. On the side of the computation analysis, for the first time, the elastic modulus of the ultra-high-rise pumping concrete was predicted by the homogenization method combined with x-ray diffraction (XRD), back scattered electron energy dispersive spectroscopy (BSE-EDS), quantitative analysis method and BSE image binary analysis method on the micro-meso-macro scale. The research route is shown in Fig. 1.

2. Materials and test methods

2.1 Materials

In this paper, P.II 42.5 type ordinary Portland cement, class I fly ash, S95 superfine slag powder and superfine silica fume were used as the cementitious materials. The chemical compositions and basic properties of the cement, fly ash, slag and silica fume are shown in **Table 1**. The sand was with the fineness modulus of 2.8, mud content of 1%, apparent density of 2582 kg/m³ and maximum particle size of 4.75 mm. The gradation of the gravel was 5 to 16 mm, the mud content was 0.3%, the crushing index was 7%, and the needle flake content was 4%. A polycarboxylate-based high range water reducing admixtures (HRWRA) with the water reducing rate of 27%, PH value of 8.3, density of 1.057 g/cm³ and solid content of 15.6% was adopted. **Table 2** shows the mix ratios of the concrete for the ultra-high-rise building engineering.

2.2 Pumping project

The pumping concrete engineering test was carried out

Fig. 1 Roadmap of this paper.

on an ultra-high-rise building in Wuhan, China. The ultra-high-rise building had 125 floors above the ground with a designed height of 636 m. The pumping test was conducted using the pumping system of the ultra-high-rise building. The pumping power was provided by an ultra-high-pressure concrete pump HBT90.48.572RS with a maximum pumping pressure reaching 48 MPa. The pumping system consisted of the horizontal pumping pipe with a length of 66 m and the vertical pumping pipe with a height of 407 m. The inner diameter and outer diameter of all the pipes were 150 mm and 168 mm, respectively. The pumping flow rate was 30 m³/h, and the average flow rate was 0.47 m/s. When the pumping height was 407 m, the pumping period of the concrete in the pump pipe was 28 min. The pumping outlet pressure was in the range of 18.6 MPa to 19.8 MPa. When the flow velocity of the pumping concrete was stable, the pump pressure was 19.3 MPa. The pumping water, paste and mortar were used to pre-wet the pipes in turn before the pumping concrete test (the amount of the cementitious materials and water used in the paste and mortar were the same as that of the concrete, and the mortar also used the same HRWRA as the concrete with a content of 1%). Pre-wetting can form a stable lubricating layer near the pipe wall before pumping the concrete so as to reduce the resistance of the concrete and the pipe during initial pumping. The concrete was sampled from the inlet and outlet of the pumping pipe when the pumping pressure was stable.

Before pumping, the slump and the slump flow of the concrete were 270 mm and 700 mm. After pumping, the slump and the slump flow of the concrete were 250 mm and 600 mm.

2.3 Methods

2.3.1 Sample preparation

In this paper, four different experiments were conducted, including the static compressive elastic modulus test, XRD test, nanoindentation test and BSE-EDS test.

The concrete used in the static compressive elastic modulus test before and after the pumping was poured into mold with a dimension of 150×150×300 mm. After the standard curing (temperature of 20°C and relative humidity of 95%) of 28 d, the samples were air-dried naturally for 24 hours at 20°C at indoor environment. The flatness of the two ends of the sample was processed, then the midline was drawn on both sides of the sample

to determine the location of the dial gauges.

Like the static compressive elastic modulus test above, the concrete before and after pumping was poured into cubic samples (100×100×100 mm³) for the XRD test. After the standard curing of 28 d and natural air drying of 24 h, 50 g paste sample was drilled from the concrete, ground into powder in an agate mortar until the powder can pass through the 80 µm square-hole sieve.

The samples for the nanoindentation test were the aggregates with ITZs cut off from the concrete cubic samples. These samples were pretreated due to the high surface smoothness requirement of the test (Zhao *et al.* 2005; Han *et al.* 2012). Firstly, alcohol was sprayed on the surface of the samples to prevent further hydration. Then the samples were placed into a cold-inlaid die, poured with epoxy resin. After that, the samples were polished with 400 mesh, 800 mesh, 1200 mesh and 2500 mesh sandpaper on the grinding and polishing machine. During the grinding process, the upper and lower planes must be parallel. Then the samples were polished with 0.25 µm diamond suspension with canvas and silk cloth in turn. The surface roughness of the polished sample was analyzed by the atomic force microscopy (AFM). When the surface roughness was less than 100 nm, the polishing requirements were met. Finally, the polished samples were cleaned with anhydrous ethanol for 15 minutes in an ultrasonic cleaner to remove the polishing machine particles or sample powder adsorbed on the surface, and then the samples were dried naturally. **Figure 2** is a sample ready for the nanoindentation test with a thickness of about 7 mm.

BSE-EDS test can directly use the nanoindentation test samples without additional treatment.

2.3.2 Static compressive elastic modulus test

The macroscopic static compressive elastic modulus test of the pumping concrete was carried out in accordance with the GB50081-2002 specification, shown in **Fig. 3**. Two dial gauges were arranged 75 mm away from the end of the sample to measure the deformation under loading. The loading speed of the test was 0.3 MPa/s. In this paper, the axial compressive strength and the elastic modulus of the pumping concrete were calculated according to the arithmetic average of six measured values.

2.3.3 XRD analysis

The XRD-7000 X-ray Powder Diffraction of Shimadzu

Fig. 2 Sample for nanoindentation test.

Table 3 Mechanical properties of concrete before and after pumping.

	Before pumping		After pumping	
	f_{cp}	E	f_{cp}	E
Average value	55.6 MPa	39.9 GPa	59.6 MPa	43.3 GPa
Standard deviation	1.09	1.49	1.09	0.96

Company, Japan was used, driven by two independent axes. It can be set by the θ -2 θ linkage drive or θ and 2θ axis independently. The scanning angle range was -6° to $+163^\circ$ (2 θ). The powder to be measured was pressed on a special glass sample plate for the diffractometer, then carried out by the XRD test.

2.3.4 Nanoindentation test

The nanoindentation test was carried out by G200 nanoindentation instrument produced by Agilent Company, USA. The hardness and elastic modulus of the sample varying with depth were measured by continuous stiffness method (CSM). During the loading process, the sample first underwent the elastic deformation. With the increase of load, the sample began to undergo the plastic deformation, and the loading curve was non-linear. The unloading curve reflected the elastic recovery process of the measured object. The hardness and elastic modulus of each test point can be obtained by analyzing the loading and unloading curve. According to Oliver-Pharr principle (Wang *et al.* 2009), the hardness H and elastic modulus E of the test point can be calculated by Eq. (1) and Eq. (2).

$$H = P/A \quad (1)$$

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A}} \quad (2)$$

where P is the peak value of the corresponding curve of the maximum load, A is the contact area at the maximum load, S is the contact stiffness [$S = (dP/dh)_{h=h_{max}}$] and the upper part (elastic section) of the unloading curve is usually used for fitting analysis. β is the pressure head

correction coefficient, for Berkovich pressure head (the angle between the center line and the side is 65.35° degrees of triangular pyramid), $\beta = 1.034$ and E_r is the effective elastic modulus defined by Eq. (3).

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \quad (3)$$

where E and ν represent the elastic modulus and Poisson's ratio of the tested materials, respectively. For cement-based materials, Poisson's ratio ν is 0.25. E_i and ν_i are the elastic modulus and Poisson's ratio of the nanoindenter, respectively. For diamond nanoindenter adopted in this study, E_i and ν_i are 1141 GPa and 0.07, respectively.

In this paper, nanoindentation tests were carried out on paste, sand ITZ and gravel ITZ of the concrete samples before and after pumping. In order to avoid the influence of the adjacent aggregates on the tested area, the nanoindentation test of the ITZ was conducted on the edge of the aggregates with spacing greater than 500 μm from the adjacent aggregates. An 8×8 array was carried out in the selected marked area with the nanoindentation depth of 2000 nm and nanoindentation spacing of 15 μm . The nanoindentation depth was set to 2000 nm so that the point can be clearly observed under the Scanning Electron Microscope (SEM). The spacing of the point was set to 15 μm with the purpose of arranging the points densely enough and maximizing the chance that the points fell in the area near the ITZ.

2.3.5 BSE-EDS analysis

The nanoindentation samples were sprayed with gold directly, then tested by the BSE-EDS. The Field Emission Environment SEM (Quanta250 FEG SEM) and EDS with 130 eV resolution were used in the experiment. The point-by-point BSE and EDS analysis were performed on the nanoindentation samples. The back-scattering working environment was under a low vacuum of 60 Pa, and the accelerating voltage used was all 20 kV. The same contrast and brightness were maintained in the image acquisition of the samples in the same age period so as to ensure the consistency of the image grayscale and the repeatability of the test.

3. Experimental results

3.1 Macroscopic elastic modulus test of concrete

The uniaxial compressive strength (f_{cp}) and elastic modulus (E) of concrete before and after pumping are shown in Table 3. After pumping, the f_{cp} and E of concrete increased by 7.19% and 8.52%.

Fig. 3 Test of elastic modulus of concrete.

3.2 XRD analysis

Figure 4 is the XRD result of the paste in the pumping concrete for 28 d. The results showed that the paste contained $2\text{CaO}\cdot\text{SiO}_2$, reaction product $\text{Ca}(\text{OH})_2$ and some unreacted mineral admixture, such as mullite ($\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) and unhydrated silica (SiO_2). It was generally believed that the C-S-H gel was one type of the amorphous substance and cannot be detected by XRD (Han *et al.* 2016).

3.3 BSE-EDS results

In this paper, the atomic percentages of each nanoindentation point were determined by the energy spectrum analysis, and then the proportions of the hydration products at each point were calculated. **Figure 5** is the partial EDS result of the gravel ITZ after pumping. Point 1 was located on the fly ash, which contained the elements of O, Al and Si. Point 2 contained the elements of C, O and Ca, indicating it located on the gravel. Point 3 contained the elements of O, Al, Si and Ca, meaning it was the paste. Point 4 was the unhydrated cement particles, which contained the elements of O, Si and Ca. When the point was on the sand, only the elements of O and Si could be detected.

3.4 Analysis of unhydrated cement and porosity of paste

In the BSE image of the paste at the age of 28 d, according to the gray eigenvalues of different phases, the percentages of phases were calculated by threshold segmentation using the Avizo image analysis software.

Then the volume fractions of the porosity and the unhydrated cement particles in the sample were calculated (Qing-Lei *et al.* 2008).

In order to accurately distinguish the unhydrated cement and pore in the paste, the segmentation threshold of the unhydrated cement was set to the range 165 to 250 and the segmentation threshold of the pore was set to the range 0 to 25, indicating that the segmentation threshold of the hydration products was 25 to 165 (Kocaba *et al.* 2012). The gray distribution of pores, hydration products and unhydrated cement particles is shown in **Fig. 6** (Hu *et al.* 2017a). In order to avoid the uncertainty caused by one BSE region, 100 regions were randomly selected for test analysis for each sample with the 2000 times magnification of the scanning region. The percentage of the unhydrated particles in each region was calculated first,

Fig. 6 Gray Distribution map of paste in BSE image.

Fig. 4 XRD analysis of cement paste.

Fig. 5 EDS analysis of nanoindentation points.

and then the average value of the 100 regions was taken as the volume ratio of the unhydrated cement of the sample.

Figures 7(a) and (b) are BSE images of the paste before and after pumping. The purple part is the unhydrated cement particles after threshold segmentation. The rest are the hydration product and the pore. According to the pixel number of the unhydrated cement particles and the paste, the volume ratio of the unhydrated cement in the image area can be calculated by Eq. (4).

$$\phi_{un} = \frac{n_{un}}{n} \times 100\% \quad (4)$$

where ϕ_{un} is the volume ratio of the unhydrated cement, n_{un} is the pixel number of the unhydrated cement, and n is the pixel number of the cement paste.

The volume ratio of the unhydrated cement in paste before pumping in **Fig. 7(a)** was 12.42%, and that in paste after pumping in **Fig. 7(b)** was 4.71%. After calculating the average value of the 100 regions, the volume

ratio of the unhydrated cement before pumping was 11.78% with the standard deviation of 4.37. The volume ratio after pumping was 5.27% with the standard deviation of 3.72. Therefore, the unhydrated cement in paste decreased by 6.51% after pumping.

Figure 8 is the binarization analysis of the porosity of the paste before and after pumping. The blue part is the pore and the rest are the unhydrated cement and the hydration product. According to the pixel number of the pore and the paste, the porosity in the BSE image area can be calculated with Eq. (5).

$$\phi_p = \frac{n_p}{n} \times 100\% \quad (5)$$

where ϕ_p is the porosity of the sample, and n_p is the pixel number of the pore.

The porosity of the paste before and after pumping in **Figs. 8(a) and (b)** were 10.89% and 4.70%, respectively. The average porosities of the paste before and after pumping were 11.73% and 4.46% with standard devia-

(a) Before pumping

(b) After pumping

Fig. 7 Binarization analysis of unhydrated cement.

(a) Before pumping

(b) After pumping

Fig. 8 Binarization analysis of paste porosity.

tions of 1.23 and 1.28, respectively. Hence, after pumping, the porosity of the paste decreased by 7.27% (= 11.73% - 4.46%) and the matrix became more compact.

3.5 ITZ thickness of pumping concrete

When the cement grains encounter the "wall" of the aggregate, a region of higher porosity near the aggregate surface will appear, due to the "packing" constraints imposed by the aggregate surface (Li *et al.* 2017). In this paper, the thickness of the ITZ was determined by analyzing the porosity of the ITZ in the BSE image. For each sample, 30 ITZs were randomly selected, and the average value of the porosity was considered as the value of the ITZ. **Figure 9** is the BSE image of one sand ITZ before and after pumping. The paste was divided into six stripes with a width of 7.5 μm along the sand edge, numbered as I, II, III, IV, V and VI in turn. Then the BSE image binarization method was used to perform the threshold segmentation for the pores in the six regions. The porosity of each region was calculated by Eq. (5).

Figure 9(a) is the porosity analysis of the sand ITZ before pumping. The porosities decreased with the increase of the distance to the edge of the sand. The porosities in area V and VI were close to the average porosity (11.73%) of the paste before pumping. The porosities of the sand ITZ after pumping is shown in **Fig. 9(b)**. The porosity of the area IV, V and VI was close to the average porosity (4.46%) of the paste after pumping. Therefore, the porosity of the sand ITZ after pumping was quite different from that before pumping.

According to the average values of the porosities in areas I, II, III, IV, V and VI of 30 samples, the porosity curves of the sand ITZ before and after pumping were drawn in **Fig. 10**. When the slope of the curve was close to 0, the porosity of the corresponding point was close to the paste, meaning that the point was already in the paste instead of the ITZ.

The porosity curves before and after pumping can be divided into two parts: the descending section and the horizontal section. The descending section corresponded

to the ITZ, and the horizontal section indicated that the porosity in this area was the same as that of the paste. The linear expression of the descending section was obtained by linear fitting the points of the descending section, and the value of the horizontal line was the same as that of the porosity of the paste. When the descending section intersected with the horizontal section, the ITZ of the aggregate can be considered to have ended (Gao *et al.* 2013), which meant the thickness of the ITZ was the distance between the edge of the aggregate and the intersection of the descending section and the horizontal line. According to the method above, the thickness of the sand ITZ before and after pumping was 28.47 μm and 23.17 μm . In this paper, the average porosity was used to represent the porosity in the ITZ. The average porosity of the ITZ was calculated by integrating the porosity curve in the ITZ section, then divided by the thickness of the ITZ. The average porosity of the sand ITZ before pumping was 23.84% and that of the sand ITZ after pumping was 7.40%.

The same method was used to determine the porosity of the gravel ITZ of the pumping concrete, shown in **Fig. 11**.

Fig. 10 Porosity curve of sand ITZ.

(a) Before pumping

(b) After pumping

Fig. 9 Porosity analysis of sand ITZ.

The porosity curves of the gravel ITZ before and after pumping were drawn in **Fig. 12**. The thickness of the gravel ITZ was 29.69 μm before pumping and 22.78 μm after pumping. The average porosity of the gravel ITZ

Fig. 12 Porosity curve of gravel ITZ.

before pumping was 24.50%, and that of the gravel ITZ after pumping was 7.35%. It was concluded that the variation trend of the sand ITZ and the gravel ITZ before and after pumping was similar.

After pumping, the porosity of matrix and ITZ in concrete decreased. The reason was that the hydration degree of cement increased after ultra-high-rise pumping, which consumed the free water in the cement slurry and reduced the number of pores caused by the free water (Choi *et al.* 2013). On the other hand, the bubbles were easily broken and overflowed under ultra-high-rise pumping, thereby reducing the porosity in the concrete after pumping.

3.6 Nanoindentation results of paste and ITZ

The nanoindentation test was carried out on the paste and the ITZ to study the pumping effect on the elastic modulus of the concrete. **Figure 13** is a sketch map of 4×4 nanoindentation results of the paste before and after pumping. The blue part is the unhydrated cement particles and the white part is the pores. The elastic modulus

(a) Before pumping

(b) After pumping

Fig. 11 Porosity analysis of gravel IT.

(a) Before Pumping

(b) After Pumping

Fig. 13 Nanoindentation results of paste.

values of each nanoindentation point are marked on the sketch map. It was observed that the elastic modulus of the area with voids was lower than the matrix whereas the elastic modulus of the area with the unhydrated cement was higher than the matrix (Constantinides and Ulm 2004). The maximum elastic modulus of the nanoindentation point before pumping was 47.1 GPa and the minimum was 18.7 GPa, indicating the average value was 30.9 GPa with the standard deviation of 8.2. After pumping, the maximum elastic modulus of the nanoindentation point was 47.4 GPa and the minimum was 20.4 GPa. Therefore, the average value was 34.0 GPa with the standard deviation of 6.7. Compared with before pumping, the average elastic modulus of the paste after pumping increased by 10.03%.

Figure 14 is the nanoindentation map of the sand ITZ of the pumping concrete. The elastic modulus of the sand was in the range 50 to 77 GPa. The average elastic modulus of the sand was 65.6 GPa. It can be seen from **Fig. 14** that the elastic modulus of the ITZ (dark gray)

was lower than the sand aggregate (blue) and the paste (light gray). The minimum and maximum elastic moduli of the ITZ before pumping [**Fig. 14(a)**] were 12.9 GPa and 27.7 GPa with the average value of 19.7 GPa and the standard deviation of 4.1. After pumping [**Fig. 14 (b)**], the minimum elastic modulus of the ITZ was 17.7 GPa and the maximum value was 35.3 GPa. The average value was 26.1 GPa and the standard deviation was 4.6. Therefore, the average elastic modulus of the ITZ after pumping was higher than that before pumping.

Figure 15 is the nanoindentation map of the gravel ITZ of the pumping concrete. The elastic modulus of the gravel was within the range 35 to 55 GPa, and the average elastic modulus of the gravel was 41.9 GPa. The minimum elastic modulus of the ITZ before pumping [**Fig. 15(a)**] was 12.8 GPa and the maximum value was 27.2 GPa with an average value of 20.0 GPa and a standard deviation of 4.2. The minimum elastic modulus of the gravel ITZ after pumping [**Fig. 15(b)**] was 19.0 GPa and the maximum value was 35.9 GPa with a mean

(a) Before Pumping

(b) After Pumping

Fig. 14 Nanoindentation results of sand ITZ.

(a) Before Pumping

(b) After Pumping

Fig. 15 Nanoindentation results of gravel ITZ.

Table 4 Atomic ratio of nanoindentation points (%).

Points	Before Pumping				After Pumping			
	PI ₁₂	PI ₂₂	PI ₂₄	PI ₃₁	PII ₁₁	PII ₂₁	PII ₂₂	PII ₄₄
O	64.31	67.94	68.38	68.59	66.17	67.84	68.62	68.82
Si	10.49	14.40	11.93	9.98	16.30	16.05	13.61	13.77
Ca	22.30	11.68	14.5	17.99	7.52	10.76	16.06	13.74
Al	2.89	5.98	5.19	3.43	10.01	5.35	1.70	3.67

Table 5 Physical properties of compositions of paste.

Composition	Molar mass (g/mol)	Density (g/cm ³)	Bulk modulus (GPa)	Shear modulus (GPa)
3Al ₂ O ₃ ·2SiO ₂ (Botero <i>et al.</i> 2012)	426	3.16	63.62	40.39
SiO ₂ (Gregorová <i>et al.</i> 2015)	60	2.20	32.60	19.10
Ca(OH) ₂ (Toropov and Leszczynski 2006)	74	2.24	40.79	17.75
3CaO·2SiO ₂ ·5H ₂ O (Haecker <i>et al.</i> 2005)	378	2.65	17.64	11.10
2CaO·SiO ₂ (Constantinides and Ulm 2004)	172	2.31	42.50	15.70
Pores	0	0.00	0.00	0.00

value of 27.0 GPa and a standard deviation of 4.8. Therefore, the elastic modulus of the gravel ITZ increased obviously after the ultra-high-rise pumping behavior.

To sum up, the elastic moduli of the paste, sand ITZ and gravel ITZ increased after ultra-high-rise pumping by 14.2%, 32.5% and 35.0%. In addition, it was observed that the ultra-high-rise pumping effect on the elastic modulus of the ITZ was much higher than that of the paste.

The air bubbles and free water in the concrete were easily broken and migrated under ultra-high-rise pumping. Because the content of air bubbles and free water in the ITZ was much higher than that in the paste, the reduction rate of ITZ porosity after pumping was greater than that of the paste. The elastic modulus of ITZ and paste increased with the decrease of porosity, so the increase of elastic modulus of ITZ after pumping was much larger than that of paste.

3.7 Compositions analysis

Ulm *et al.* (2007) used statistical nanoindentation analysis to determine the relative volume fraction of the phase in cement-based materials. However, using this method to calculate the volume fraction of phase in ITZ requires tremendous experiments. In this paper, based on the EDS test results of matrix and ITZ, the volume fraction of each phase was calculated, then the elastic modulus was calculated by homogenization method, and finally compared with the nano indentation test results to verify the reliability of the calculation results.

Through the XRD analysis, it was confirmed that there were Ca(OH)₂, 2CaO·SiO₂, 3Al₂O₃·2SiO₂ and SiO₂ in the paste of the pumping concrete. There were certainly a large number of the C-S-H gels in the hydration products. According to literature (L'Hôpital *et al.* 2016), the chemical equation of the C-S-H gel in this paper was determined as 3CaO·2SiO₂·5H₂O. The atomic percentages of the nanoindentation points in the paste and the ITZ were obtained by the BSE-EDS test, and then the

volume fractions of the phases at those points were calculated. Among them, the volume ratios of the unhydrated cement particles and pore were directly calculated by binarization analysis of the BSE image. After removing the corresponding element content of the unhydrated cement particles, the molecular ratios of the other four components were calculated by establishing a linear equation system with four unknowns (Li *et al.* 2017), as shown in Eq. (6).

$$\begin{bmatrix} 13 & 2 & 2 & 12 \\ 2 & 1 & 0 & 2 \\ 0 & 0 & 1 & 3 \\ 6 & 0 & 0 & 0 \end{bmatrix} \times \begin{bmatrix} x_1 \\ x_2 \\ x_3 \\ x_4 \end{bmatrix} = \begin{bmatrix} R_O \\ R_{Si} \\ R_{Ca} \\ R_{Al} \end{bmatrix} \quad (6)$$

where x_1 , x_2 , x_3 and x_4 represent the molecular percentages of the four components, which are 3Al₂O₃·2SiO₂, SiO₂, Ca(OH)₂ and 3CaO·2SiO₂·5H₂O, respectively. R_O , R_{Si} , R_{Ca} and R_{Al} represent the atomic percentage of four elements (O, Si, Ca and Al). The coefficients of the equation correspond to the atomic contents of the corresponding elements in the four chemical equations. For example, in the first row of the coefficient matrix, 13 represents that there are 13 O atoms in 3Al₂O₃·2SiO₂, 2 represents that there are two O atoms in SiO₂, 2 represents that there are two O atoms in Ca(OH)₂ and 12 represents that there are 12 O atoms in 3CaO·2SiO₂·5H₂O.

After obtaining the molecular ratios of components of the nanoindentation point, the mass ratio of the four components were calculated based on the molar mass of each component. In the case of determining the density of each component, the volume fraction of the nanoindentation point can be calculated by dividing the mass of each component by its density. **Table 4** lists the atomic percentages of the eight nanoindentation points of the paste before and after pumping in **Fig. 13**. **Table 5** lists the molar mass, density and related mechanical properties of each component.

Table 6 Volume fractions of compositions on nanoindentation points (%).

Points	Before Pumping				After Pumping			
	PI ₁₂	PI ₂₂	PI ₂₄	PI ₃₁	PII ₁₁	PII ₂₁	PII ₂₂	PII ₄₄
3Al ₂ O ₃ ·2SiO ₂	6.83	13.68	14.03	8.85	24.40	13.55	4.30	8.66
SiO ₂	3.11	15.54	8.08	4.17	27.95	24.81	19.40	12.18
Ca(OH) ₂	25.26	4.93	11.52	25.53	7.98	5.46	22.21	4.50
3CaO·2SiO ₂ ·5H ₂ O	28.09	49.45	66.37	61.45	27.33	49.80	54.09	62.15
2CaO·SiO ₂	36.71	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Pores	0.00	16.40	0.00	0.00	12.34	6.37	0.00	12.51

Table 7 Volume fractions of compositions of paste (%).

Compositions	Paste		Sand ITZ		Gravel ITZ	
	Before	After	Before	After	Before	After
3Al ₂ O ₃ ·2SiO ₂	12.36	11.25	11.76	5.16	10.52	6.38
SiO ₂	10.13	14.23	6.69	7.41	6.56	8.46
Ca(OH) ₂	9.61	10.66	8.03	5.26	8.83	6.12
3CaO·2SiO ₂ ·5H ₂ O	44.39	54.69	50.39	74.82	50.81	72.10
2CaO·SiO ₂	11.78	4.71	0.00	0.00	0.00	0.00
Pores	11.73	4.46	23.13	7.35	23.28	6.94

The volume fractions of the 2CaO·SiO₂ and pores at the nanoindentation point were determined by the BSE binarization analysis. After removing the atomic percentage of the 2CaO·SiO₂, the volume fractions of the other four substances were calculated. **Table 6** is the volume fractions of the 6 constituents of the 8 nanoindentation points in **Fig. 13**.

According to the analysis results of 128 nanoindentation points in the paste before and after pumping, the volume fractions of four components (3Al₂O₃·2SiO₂, SiO₂, Ca(OH)₂ and 3CaO·2SiO₂·5H₂O) in paste before and after pumping were taken as the average of 64 nanoindentation points, respectively, and the contents of the 2CaO·SiO₂ and pores were determined by the BSE image binarization in Section 3.2. The calculation method for the volume fraction of each component in the ITZ was the same as the paste. The volume contents of the components in the paste and the ITZ before and after pumping are shown in **Table 7** (Hu *et al.* 2017b). After pumping, the C-S-H in the paste increased significantly by 10.3%, the SiO₂ and Ca(OH)₂ increased slightly, while the 3Al₂O₃·2SiO₂ decreased slightly. Compared with the paste, the content of the C-S-H in the ITZ increased, and the content of the C-S-H in the ITZ after pumping was up to 70%. There were no unhydrated cement particles in the ITZ.

4. Evaluation of elastic modulus of concrete by homogenization

The static compressive elasticity modulus test of concrete can only measure the modulus of elasticity of concrete at macro level, while the effect of ultra-high-rise pumping on the mechanical properties and components of concrete was mainly reflected at micro level. The homogenization method can be used to calculate the micro, meso and macro elastic modulus of the ultra-high-rise pumping concrete, then studied the influence of the ultra-high-rise pumping on the micro, meso

and macro elastic modulus of the concrete.

In this section, the elastic moduli of the paste and ITZ before and after pumping were calculated by homogenization method of which the reliability was verified by comparing with the results of the nanoindentation on the micro scale. Then the Lu and Torquato model was used to calculate the volume fraction of each phase of the pumping concrete on the meso and macro scales. Afterward, the homogenization method was used to predict the elastic modulus of the concrete after ultra-high-rise pumping on the meso and macro scales. Combining the BSE-EDS test with the Lu and Torquato model, the validity of the homogenization method for calculating elastic modulus of the ultra-high-rise pumping concrete was verified, and the pumping influence on the elastic modulus of the concrete on micro-meso-macro scales was clarified.

4.1. Homogenization method

For concrete materials, Self-Consistent (SC) was a common homogenization method (Budiansky 1965; Hill 1965). This method did not need to distinguish the difference in morphology between inclusions and matrix. That is to say, this method was suitable for composite materials whose reference medium was difficult to determine. According to the multi-scale method proposed by Ulm (Constantinides and Ulm 2004) and the characteristics of the hydration products in this paper, concrete can be divided into three levels, as shown in **Fig. 16**. After determining the volume fraction of each component in the paste and the elastic modulus of the ITZ, the elastic modulus of the concrete after pumping can be predicted by the SC method on micro-meso-macro scale.

In the first scale (Level I), the paste contained C-S-H gel, CH crystal, unhydrated cement particle, SiO₂, 3Al₂O₃·2SiO₂, and pore with the characteristic length range 10⁻⁶ to 10⁻⁴ m. On this scale, it was difficult to distinguish the matrix from the inclusion due to the complexity of each component at the nanoindentation

point and the variability of the volume fraction. Therefore, the elastic modulus of the nanoindentation point was calculated by the SC method (Hill 1965). According to the bulk modulus, shear modulus and volume fraction of each component at each nanoindentation point in **Table 5**, the bulk modulus and shear modulus of each nanoindentation point on the paste of the pumping concrete can be calculated by Eqs. (7) and (8).

$$k^{\text{hom}} = k_0 + \sum_{r=1}^N \frac{c_r(k_r - k_0)(3k^{\text{hom}} + 4\mu^{\text{hom}})}{3k_r + 4\mu^{\text{hom}}} \quad (7)$$

$$\mu^{\text{hom}} = \mu_0 + \sum_{r=1}^N \frac{5c_r\mu_r^{\text{hom}}(\mu_r - \mu_0)(3k^{\text{hom}} + 4\mu^{\text{hom}})}{3k^{\text{hom}}(3\mu^{\text{hom}} + 2\mu_r) + 4\mu^{\text{hom}}(2\mu^{\text{hom}} + 3\mu_r)} \quad (8)$$

where k^{hom} and μ^{hom} represent bulk modulus and shear modulus, respectively. The elastic modulus E^{hom} of the paste and the ITZ can be calculated by Eq. (9).

$$E^{\text{hom}} = \frac{9k^{\text{hom}}\mu^{\text{hom}}}{3k^{\text{hom}} + \mu^{\text{hom}}} \quad (9)$$

The second scale (Level II) regarded the paste as the matrix phase of the mortar, ITZ and sand aggregate as the inclusions. The elastic moduli of the paste and ITZ were calculated using the above calculation results from Level I. The nanoindentation test value of the sand was used as the elastic modulus of the sand aggregate in this paper (65.6 GPa). The elastic modulus of the mortar was calculated by the SC method [Eqs. (7) and (8)].

The third scale (Level III) took the mortar as the matrix phase of the concrete, the ITZ and gravel aggregate as the inclusions. The elastic moduli of the mortar and the ITZ were calculated from Level II, and the elastic modulus of the gravel was measured by the nanoindentation test (41.9 GPa). Then the SC method [Eqs. (7) and

Fig. 16 Multiscale microstructure of concrete.

(8)] was used to calculate the elastic modulus of the pumping concrete.

4.2 Calculation of volume fraction of phases

The volume fraction of the sand in the mortar can be calculated according to the design value of the mix ratio of the pumping concrete, Eq. (10).

$$f_s = \frac{v_s}{v_s + v_w + v_c + v_{fa} + v_{sf} + v_{sp}} \quad (10)$$

$$= \frac{m_s / \rho_s}{m_s / \rho_s + m_w / \rho_w + m_c / \rho_c + m_{fa} / \rho_{fa} + m_{sf} / \rho_{sf} + m_{sp} / \rho_{sp}}$$

where f_s is the volume fraction of the sand in the mortar, v_s is the volume of the sand in the mortar, v_w , v_c , v_{fa} , v_{sf} and v_{sp} are the volumes of the initial water, cement, fly ash, silica fume and micro-mineral powder in the mortar, m_w , m_c , m_{fa} , m_{sf} and m_{sp} are the mass of the water, cement, fly ash, silica fume and micro-mineral powder. ρ_s , ρ_w , ρ_c , ρ_{fa} , ρ_{sf} and ρ_{sp} are the density of the water, cement, fly ash, silica fume and micro-mineral powder, respectively.

According to the mix ratio of the mortar and the density of the sand aggregate, water, cement, fly ash, silica fume and micro-mineral powder ($\rho_s = 2.62 \text{ g/cm}^3$, $\rho_w = 1.00 \text{ g/cm}^3$, $\rho_c = 3.10 \text{ g/cm}^3$, $\rho_{fa} = 2.78 \text{ g/cm}^3$, $\rho_{sf} = 2.20 \text{ g/cm}^3$, $\rho_{sp} = 2.81 \text{ g/cm}^3$), the volume fraction of the sand aggregate in the mortar sample was calculated as $f_s = 51.19\%$.

The Lu and Torquato model was used to calculate the volume fraction of the ITZ (Garboczi and Bentz 1997), as shown in **Fig. 17**. The overlapping of the ITZ between the adjacent aggregates was considered in this model. Its basic assumption was that the sand aggregate was a circular particle and the ITZ was a shell with a thickness of t , wrapping around the circular sand aggregate. The volume fraction of the cement paste was considered as the left area except the sand aggregate and the ITZ.

When the model was applied to the mortar, the volume fraction of the cement paste matrix can be defined as $e_v(t)$, assuming that the spherical particles of different scales were randomly distributed in the cement paste matrix. Thus, if the thickness of the ITZ was infinite, i.e., $t \rightarrow \infty$, the area occupied by the paste in the mortar tended to be 0. Based on the above assumptions, the volume fraction of the ITZ can be expressed as:

$$f_{\text{ITZ}} = 1 - f_s - e_v(t) \quad (11)$$

Fig. 17 Schematic diagram of the Lu and Torquato model.

Table 8 Calculated elastic modulus (GPa) of nanoindentation points.

PI _{ij}	Before Pumping				PII _{ij}	After Pumping			
	PI _{i1}	PI _{i2}	PI _{i3}	PI _{i4}		PII _{i1}	PII _{i2}	PII _{i3}	PII _{i4}
PI _{1j}	46.3	40.8	33.8	32.6	PII _{1j}	36.5	43.7	44.9	32.1
PI _{2j}	34.6	25.4	22.4	36.3	PII _{2j}	34.1	36.4	36.0	33.4
PI _{3j}	35.9	21.6	22.6	28.9	PII _{3j}	35.5	34.8	27.5	34.4
PI _{4j}	34.4	35.5	21.9	17.5	PII _{4j}	19.6	36.2	28.1	25.5

The following equation was obtained by substituting the thickness t of the ITZ and the size distribution of the sand aggregate.

$$e_v(t) = (1 - f_s) \exp \left\{ -2f_s S \left[a_0 \left(\frac{t}{D_N} \right)^3 + a_1 \left(\frac{t}{D_N} \right)^2 + a_2 \left(\frac{t}{D_N} \right) \right] \right\}, \quad t \geq 0 \quad (12)$$

where t is the thickness of the ITZ, $a_0 = [4B(1 - f_s)(1 - f_s + 3f_s S) + 4Af_s^2 S^2] / (1 - f_s)^3$, A is a constant, usually 0, 2 or 3. Assuming that the sand aggregate is circular, $A = 0$ is the best choice, $a_1 = [6B(1 - f_s) + 9f_s S] / (1 - f_s)^2$; $a_2 = 3 / (1 - f_s)$, $B = (\overline{D_N})^2 / \overline{D_N^3}$, $S = (\overline{D_N^2} \times \overline{D_N})^2 / \overline{D_N^3}$, $\overline{D_N}$ is the average diameter of aggregate quantity, $\overline{D_N^2}$ is the second-order origin moment of the average diameter of aggregate quantity and $\overline{D_N^3}$ is the third-order origin moment of the average diameter of aggregate quantity.

The volume fraction of the paste in the mortar can be calculated according to the following equation:

$$f_c = 1 - f_s - f_{ITZ} \quad (13)$$

The thickness of the sand ITZ before pumping was 28.47 μm . The volume fractions of the ITZ and the paste in the mortar before pumping were 20.42% and 28.39%, respectively by using Eqs. (10), (11), (12) and (13). The thickness of the sand ITZ after pumping was 23.17 μm , so the volume fraction of the ITZ and the paste were 16.12% and 32.69%, respectively.

The density of the gravel was 2.70 g/cm^3 . The volume fraction of the coarse aggregate in the pumping concrete was 37.58% by using the same calculation method with Eq. (10). The thickness of the gravel ITZ before pumping was 29.69 μm , and the volume fractions of the ITZ and the mortar in the concrete before pumping were calculated by Eqs. (11), (12) and (13), which were 1.35% and 61.07%, respectively. After pumping, the thickness of the gravel ITZ was 22.78 μm , and the volume fractions of the ITZ and the mortar were 1.16% and 61.25%, respectively.

4.3 Calculation of elastic modulus of pumping concrete

According to Eq. (6), the volume fraction of each component at each nanoindentation point in Fig. 13 was calculated, and then the elastic modulus of the paste in the pumping concrete, i.e., the elastic modulus of Level I, was calculated by Eqs. (7) and (8). The calculated elastic moduli of partial nanoindentation points before and after

pumping are shown in Table 8.

In Table 8, the calculated elastic moduli of the paste before and after pumping were close to that from the nanoindentation test (Fig. 13). The average relative errors of the elastic modulus of the paste before and after pumping were 3.98% and 3.89%, respectively. According to the volume fraction of each component in Table 7, the elastic modulus of the paste (Level I) before and after pumping was calculated by Eqs. (7), (8) and (9), which were 29.6 GPa and 33.9 GPa, respectively.

The elastic modulus of the ITZ of the pumping concrete in Table 7 was calculated by Eqs. (7) and (8). The calculated elastic modulus of the sand ITZ before pumping was 19.2 GPa with a relative error of 2.54%, compared with the results from the nanoindentation test. The calculated elastic modulus of the sand ITZ after pumping was 26.9 GPa with a relative error of 3.07%. The calculated elastic modulus of the gravel ITZ before pumping was 18.8 GPa with a relative error of 6.00%. The calculated elastic modulus of gravel ITZ after pumping was 27.9 GPa with a relative error of 3.33%.

According to the test results of the nanoindentation, the elastic modulus of the sand was 65.6 GPa. Besides, the Poisson's ratio of the sand was 0.15 (Li *et al.* 1999). The shear modulus and bulk modulus of the sand calculated by Eqs. (14) and (15) were 28.5 GPa and 31.2 GPa, respectively.

$$\mu = \frac{E}{2(1 + \nu)} \quad (14)$$

$$k = \frac{E}{3(1 - 2\nu)} \quad (15)$$

According to the homogenized elastic modulus calculation results of the paste mentioned above (Level I), the shear modulus and bulk modulus of the paste before pumping were calculated using Eqs. (14) and (15) as 11.8 GPa and 19.7 GPa, respectively, with the Poisson's ratio of the paste being 0.25 (Li *et al.* 1999). The shear modulus and bulk modulus of the paste after pumping were 13.6 GPa and 22.6 GPa. The shear modulus and bulk modulus of the sand ITZ before pumping were 7.4 GPa and 16.0 GPa, respectively according to Eqs. (14) and (15), and the shear modulus and bulk modulus of the sand ITZ after pumping were 10.3 GPa and 22.4 GPa, respectively, and the Poisson's ratio of the ITZ was 0.3 (Li *et al.* 1999).

Based on the volume fraction of the paste, ITZ and sand in the pumping concrete in Section 4.2, the elastic moduli of the mortar (Level II) before and after pumping

Table 9 Calculation results of elastic modulus of concrete before and after pumping.

	Theoretical Value	Experimental Value	Relative Errors
Before Pumping	40.8 GPa	39.9 GPa	2.26%
After Pumping	44.2 GPa.	43.3 GPa	2.08%

were calculated by using Eqs. (7) and (8), which were 40.7 GPa and 46.1 GPa, respectively.

The elastic modulus of the gravel from the nanoindentation test was 41.9 GPa. The shear modulus and bulk modulus of the gravel calculated by Eqs. (14) and (15) were 17.5 GPa and 23.3 GPa, respectively, and the Poisson's ratio of the gravel was 0.20 (Li *et al.* 1999). According to the calculation results of the elastic modulus of the mortar in Level II, the shear modulus and bulk modulus of the mortar before pumping were 16.8 GPa and 23.4 GPa, while the shear modulus and bulk modulus of the mortar after pumping were 19.0 GPa and 26.5 GPa respectively and the Poisson's ratio of mortar was 0.21 (Li *et al.* 1999). According to the calculation results of the homogenization method for elastic modulus of the gravel ITZ in the pumping concrete, the shear modulus and bulk modulus of the gravel ITZ before pumping were 7.2 GPa and 15.7 GPa, respectively. Those two values of the ITZ after pumping were 10.7 GPa and 23.2 GPa, respectively.

According to the volume fraction, the shear modulus and the bulk modulus of the mortar, ITZ and gravel in Level III, the elastic modulus of the pumping concrete was predicted by the homogenization method [Eqs. (7) and (8)]. The calculation results of elastic modulus of concrete before and after pumping are shown in **Table 9**. The elastic modulus of the concrete before pumping was 40.8 GPa and after pumping, that was 44.2 GPa. The relative errors between the experimental and theoretical values of the elastic modulus of the concrete samples before and after pumping were 2.26% and 2.08%, respectively.

The calculated results of the elastic modulus of the paste and ITZ obtained by combining the BSE image analysis method with the SC method were of high accuracy to those obtained by the nanoindentation test. Then the elastic modulus of the ultra-high-rise pumping concrete can be predicted promisingly on multi-scale by combining the SC method with the Lu and Torquato model.

The homogenization method was used to calculate accurately the elastic modulus of concrete at micro, meso and macro scales before and after pumping. The relationship between the micro elastic modulus and the macro elastic modulus of the concrete was established, and the influence of the ultra-high-rise pumping action on the concrete elastic modulus at micro, meso and macro scales was accurately analyzed.

5. Conclusions

Based on the engineering test of the pumping concrete, the pumping influence on the composition and micro-structure of the concrete was studied. The elastic

modulus of the pumping concrete was calculated by the homogenization method on multi-scales. The following conclusions are drawn from the test and the analysis:

- (1) After pumping with a vertical height of 407 m and a maximum pumping pressure of 19.3 MPa, the axial compressive strength and the macroscopic elastic modulus of concrete increased by 7.29% and 8.52%, respectively.
- (2) The BSE image binarization method was used to analyze the ultra-high-rise pumping concrete. After pumping, the porosity of the paste decreased by 7.27% and the unhydrated cement decreased by 6.51%. The influence of the pumping process on the sand ITZ and the gravel ITZ in concrete was similar. The thickness of the sand ITZ after pumping decreased by 5.3 μm and the average porosity of the ITZ decreased by 16.44%. The thickness of the gravel ITZ decreased by 6.91 μm and the average porosity of the ITZ decreased by 17.15%. The porosity of the ITZ decreased significantly after pumping.
- (3) According to the nanoindentation test results, the elastic moduli of the paste and the ITZ of the pumping concrete increased due to the ultra-high-rise pumping behavior. Compared with before pumping, the elastic modulus of the paste after pumping increased by about 10.03%, that of sand ITZ and gravel ITZ increased by 32.5% and 35.0%. The effect of the ultra-high-rise pumping on the elastic modulus of the ITZ was much higher than the paste.
- (4) According to the BSE image analysis method and the EDS test results, the combination of the SC method and the Lu and Torquato model can accurately predict the elastic modulus of the concrete after the ultra-high-rise pumping on the micro-meso-macro scale.

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