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In-situ micro-CT characterization of mechanical properties and failure mechanism of cementitious syntactic foams

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ABSTRACT

The advancements in structural materials are guided by the desire of lowering the density and increasing the strength. Composite materials show promise in tuning the density and strength to meet specific design requirements. Lightweight cementitious materials, such as foamed concretes, are generally known to show poor mechanical properties (e.g., compressive strength and elastic modulus). The lack of control over the size, shape, and distribution of air voids severely limits the improvement of mechanical properties in lightweight cementitious materials. This work is focused on manufacturing and examining the mechanical properties of cementitious syntactic foams with hollow glass microspheres. Use of hollow particles to incorporate porosity allows for the control over the size, shape, and volume fraction of voids present in the composite. Hollow glass microspheres with several different densities (0.15–0.60 g/cm^3) are used in different volume fractions (20%–50%) to manufacture the cementitious syntactic foams. The results show that cementitious syntactic foams (CSF) have compressive strengths (32–88 MPa) and elastic moduli (10–20 GPa) for a given range of low density (1.15–1.80 g/cm^3), which are better than other cellular cementitious materials in the same density range. In-situ micro-CT scan results reveal that the micro-fracture mechanisms in CSFs under compressive loading depend on the microsphere density and aging of the material.

1. Introduction

Low density and high compressive strength are desired properties for structural materials. Lightweight materials reduce the self-weight of the structures, which ultimately leads to improved earthquake performance and reduced foundation costs [1]. Lightweight cementitious materials could be manufactured through either lightweight aggregates or a cellular structure. However, reducing the proportion of the strong matrix material inevitably leads to a reduction in strength and stiffness. Cellular cementitious composites such as foamed concretes are commonly used for their low cost, light weight, high heat capacity, excellent fire resistance, and moderate thermal insulation properties [2–5]. These properties make them useful for precast blocks, precast wall elements, prefabricated insulation boards, in-situ void fills, trench reinstatements, and lightweight aggregates for lightweight concretes. However, foamed concretes are not commonly used for structural applications as they have poor mechanical properties. In this study, a type of particulate composite called cementitious syntactic foam (CSF) is studied as a lightweight material with improved mechanical properties in comparison to foamed concretes.

It is important to note that, foamed concrete is a type of composite where the cement paste or the mortar is introduced with small air voids to create a lightweight material. These air voids are either generated during the wet mixing procedure or pre-formed as a foam and later incorporated into the wet mix. When foamed concretes with the same void ratio are considered, it has been shown that materials with smaller sized and closely spaced air voids show higher compressive strengths than materials with larger sized and farther spaced air voids [6–8]. Larger sized and farther spaced air voids in foamed concretes are usually created when smaller sized air voids come into contact and merge [9–11]. Consequently, it is difficult to limit the maximum size of the air voids as the void ratio of the foamed concrete increases. Therefore, foamed concretes have low compressive strengths (1–10 MPa) and elastic moduli (1–8 GPa) in comparison to conventional concrete.

Furthermore, it is difficult to design and manufacture foamed

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concretes for a specific dry density, since foamed concretes desorb varying amounts of the total mix water depending on several factors such as concrete wet density, early curing, and subsequent environmental conditions [12,13]. However, it is much easier to design and manufacture CSFs with a specific density.

In an effort to achieve higher compressive strengths and elastic moduli, while maintaining desired properties of foamed concretes such as low density, this study proposes a set of particulate composites called CSFs. These lightweight composites are manufactured with hollow glass microspheres (HGM) dispersed in the cementitious matrix instead of air voids. The particulate structure of the CSFs ensures a better control over the void ratio, void size distribution, and void shapes. The HGMs of CSFs prevent merging of the voids and these HGMs can easily be sieved to achieve a desired void size distribution. Therefore, a particular void ratio can be achieved through CSFs with smaller sized voids (2–100 μm) than foamed concretes (100–1000 μm) [9]. Likewise, in contrast with foamed concretes, CSFs, with an optimum HGM size distribution, can be manufactured to achieve high void ratios and low densities without transforming into an open cell structure.

In the past, cementitious particulate composites have been designed and manufactured with cenospheres which are contained in the fly ash from coal power stations [14–16]. However, only a small portion of the fly ash is cenospheres (~5% by weight) and in comparison to commercially available HGMs, cenospheres usually have higher true densities ($\geq$0.99 g/cm^3) and larger particle sizes (0.10–2.00 mm).

In this study, five different commercially available HGMs are used with different volume fractions to manufacture the CSFs. Density measurements, scanning electron microscopy and micro-CT scanning are utilized to investigate the effects of the HGM density and the volume fraction on the material microstructure. All of these CSFs are then tested under quasi-static loading for their compressive strengths and a selected set of CSFs are tested for their elastic moduli.

It is essential to remember that the crack propagation mechanisms at the microstructural level significantly affect the strength of a composite material. Improving the properties of micro-composites, without an understanding of the microstructural mechanisms that lead to the material failure, is difficult. The macro-mechanical behavior of the composite materials is a result of the interaction between the matrix material, the inclusions and the interfacial zone between them. It has been shown that cementitious composites, such as concrete, have a complex crack propagation mechanism at the microscale [17]. Consequently, a close investigation of the micro-fracture mechanism of the composite material throughout all the loading stages is necessary. Therefore, an in-situ compressive test is performed inside a micro-CT scanner to examine the micro-fracture mechanisms of CSFs with different density HGMs.

2. Materials, manufacturing and methods

2.1. Materials and mix proportions

The CSFs studied in this work were manufactured with a Type V White Portland cement from Aalborg, Denmark without the use of any supplementary cementitious materials. The chemical and mineral compositions of Type V White Portland cement are reported in Table 1. The rheology of the matrix material was adjusted with MasterGlenium 7500 full-range water-reducing admixture (HRWR), a polycarboxylate ether-based superplasticizer (PCEs) manufactured by BASF™.

In order to investigate the impact of microsphere size and density, different HGMs made of soda-lime-borosilicate glass, supplied by 3M™, were used to manufacture the CSFs. The HGMs were labeled with a letter followed by a two-digit number, where the letter indicates the series and the two-digit number refers to the nominal true particle density of the microsphere. For example, K46 represents series K HGMs with a density of 0.46 g/cm^3.

Table 1
Chemical and mineral composition of the cement.

	Composition	wt. %
Chemical composition	Calcium oxide, CaO	67.19
	Silicon dioxide, SiO_2	24.59
	Aluminum oxide, Al_2O_3	2.09
	Iron oxide, Fe_2O_3	0.42
	Magnesium oxide, Na_2O	0.70
	Total Alkalinity as $Na_2O+0.658K_2O$	0.19
	Sulfur oxide, SO_3	2.23
	Insoluble Residue	0.09
	Loss on Ignition (LOI)	1.41
Mineral composition	Tricalcium Silicate, C_3S	63.85
	Dicalcium Silicate, C_2S	22.33
	Tricalcium Aluminate, C_3A	4.83
	Tetracalcium Aluminoferrite, C_4AF	–

This study has used five different HGMs K15, S22, S32, K46, and S60 (Table 2). S and K series denote a change in the variation of the size distribution, where K series has a wider particle size distribution. The isostatic crush strength increases significantly with the increase in density of the HGMs, e.g., it increases by ~25 times from S22 to S60 as specified by the manufacturer. The particle size distribution of these HGMs is specified by the D_{10}, D_{50} and D_{90} values by 3M™, which represents the cumulative 10%, 50%, and 90% finer HGM sizes, respectively. Additionally, a QICPIC particle size analyzer (Sympatec™) is used to measure particle size distribution of all types of HGMs (Fig. 1-a). The results indicate that lower density particles are relatively larger in diameter compared to the higher density particles. Table 2 lists the comparison of D_{10}, D_{50}, and D_{90} values between QICPIC measured (meas.) and values specified by 3M™ (spec.) for all the five cases.

Scanning electron microscope (SEM) image, shown in Fig. 1-b, confirms the spherical shape and the hollow structure of the HGMs. The average diameter of HGMs ranges from 30 to 60 μm. From Fig. 1-a, it is clear that finding different density HGMs with similar particle size distribution is difficult since stronger and heavier HGMs also tend to be smaller in size for the selected product range. Therefore, a parameter named radius ratio η is useful in characterizing the geometrical properties of HGMs. Radius ratio η is defined as (r_i/r_o), where r_i is the inner radius and r_o is the outer radius of the HGMs. Since it is difficult to measure the inner radius for individual particles, the average η for a batch of particles can be estimated by (1).

$$\eta = \sqrt[3]{1 - \frac{\rho_b}{\rho_g}} \tag{1}$$

where, ρ_g is the glass material density of 2.54 g/cm^3, and ρ_b is the true particle density. The average wall thickness of the HGMs, t_w can also be calculated based on η as in (2).

$$t_w = (1 - \eta) r_o \tag{2}$$

While η monotonously decreases with increase in true density of the HGMs, i.e, η is larger for lighter HGMs, there seems to be no strict correlation with t_w as the average HGM sizes change with density (Table 2). Therefore, variation in material properties is better explained with η than t_w. Previous experimental studies highlighted the significant correlation between η and the