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Application of nanoindentation testing to study of the interfacial transition zone in steel fiber reinforced mortar

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ABSTRACT

The characteristics of the profiles of elastic modulus and hardness of the steel fiber–matrix and fiber–matrix–aggregate interfacial zones in steel fiber reinforced mortars have been investigated by using nanoindentation and Scanning Electron Microscopy (SEM), where two sets of parameters, i.e. water/binder ratio and content of silica fume were considered. Different interfacial bond conditions in the interfacial transition zones (ITZ) are discussed. For sample without silica fume, efficient interfacial bonds across the steel fiber–matrix and fiber–matrix–aggregate interfaces are shown in low water/binder ratio mortar; while in high water/binder ratio mortar, due to the discontinuous bleeding voids underneath the fiber, the fiber–matrix bond is not very good. On the other hand, for sample with silica fume, the addition of 10% silica fume leads to no distinct presence of weak ITZ in the steel fiber–matrix interface; but the effect of the silica fume on the steel fiber–matrix–aggregate interfacial zone is not obvious due to voids in the vicinity of steel fiber.

1. Introduction

Fiber reinforced mortar is a composite structure at a microscopic scale. Its properties rely on the matrix, aggregate, fiber and the interfacial transition zone (ITZ) between the two. The transition area consists of a loose unit of hydrates of the neighboring cement grains. It consolidates with the progressing hydration and reaches in its "final phase" a porosity of about 50% [1]. The origin of the ITZ mainly lies in the so called "wall" effect of packing of cement grains against the relatively flat aggregate surface [2] or fiber, or steel surface, micro-bleeding effect which results in the accumulation of water under the aggregate particles and the flocculation effect of the small cement grains [3].

Due to the way it is formed the ITZ is not a definite zone, but a region of transition. Its effective thickness varies with the microstructural feature being studied and during the course of hydration [2]. The typical width of the ITZ between the aggregate and matrix is 50 μm; however, different researchers obtained different thicknesses of ITZ from their tests. For instance, the thickness of the transition zone between the aggregate and matrix ranged from 10 μm to about 30 μm [1]; Ollivier et al. [3] argued that there was only thickness of 15 μm to 20 μm around the aggregates, just equaling to the mean diameter of the cement grains. For the fiber–mortar interface, where amorphous cast iron fibers were used, fiber–matrix debonding generally occurred at some distance – about 5 μm – from the fiber surface where the porous zone was the weakest [4]. Although the size of the ITZ varied with different fiber type and fiber size as well as matrix details, most observations suggested a relatively large porous and weak layer on the order of 40 μm to 70 μm thickness [5]. For the ITZ around steel reinforcement, it was shown that the minimum micro-mechanical properties occurred at 10 ± 30 μm from the actual steel interface [6].

In the study of ITZ, a key question is to what extent the existence of ITZ has any practical influences on the engineering properties of cementitious materials, or it is just a peculiarity of academic interest [7]. There are two contrary opinions about this problem: some researchers argued that the ITZ is the weakest link between the cement paste and the aggregate, so it has a significant role in determining the properties of all cementitious composites [8–10]; however, Diamond and Huang [11] pointed out, that there is no reason to assume the significant negative effects of ITZ on permeance or mechanical properties of concrete, even for concrete with a water/binder ratio of 0.5. The reason for lack of conclusive evidences provided by various experimental researches carried out to the ITZ mainly lies in the limited sensitivity of the experimental technique or inappropriate methods yielding biased information [12]. In order to resolve this issue two committees were set up by RILEM and the conclusion are as follows [7]: 1) the ITZ should be viewed as a system property which is dependent on the overall composition and the method of fabrication of the cement composite; 2) the properties of the ITZ may have a moderate influence on the mechanical properties of concrete but not a drastic one; 3) the ITZ may have a drastic effect on the mechanical

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Table 1
Particle size and density of the steel fiber reinforced compounds.

Material	Particle size (μm)	Density (kg/m^3)
Anlegg cement	<30 μm: 80.1% and >90 μm:0.5%	3120
Silica fume	Retained on 45 μm sieve<1.5%	2200
Limestone powder	<30 μm: 64.3% and >90 μm: 5.6%	2730
Sand 0–4 mm	<125 μm: 6.1% and <4000 μm: 98.9%	2650
Sand 0–2 mm	<125 μm: 23.6% and <2000 μm: 91.8%	2650
Steel fiber	160 (diameter)	7800
Glenium 151	–	1030
Water	–	1000

properties of fiber reinforced cement composites and on their long term properties.

During the last decades, a lot of research works have been carried out to study the influence of various factors on ITZ between aggregate and paste. The ITZ between the fiber and matrix in fiber reinforced cement based composites was also studied due to its drastic effect on the mechanical and durability properties. The ITZ between the fiber and matrix was affected by the water/binder (w/b) ratio [13], age [14] and sand content [15]. Mineral admixtures such as silica fume [4,13,16] and fly ash [17] were also introduced to improve the quality of the fiber–matrix interfacial zone.

The morphological characteristic of the microstructure of ITZ is primarily characterized via electron microscopy. Throughout those researches, Scanning Electron Microscopy (SEM) [10,11], Environmental Scanning Electron Microscopy (ESEM) [13] and Transmission Electron Microscopy (TEM) [18] have been extensively used. In order to investigate and quantify microstructural gradients across the ITZ, backscattered electron imaging and energy dispersive X-ray map [2,19,20] have been also used. However, for the above mentioned microscopical techniques, only two dimensional sections of a three-dimensional microstructure can be observed [19].

For the purpose of developing test methods that can be used to determine the mechanical characteristics of microsize zones in various cement based composites, in locations that exhibit microstructural gradients, microhardness or microindentation testing was used to characterize gradients in mechanical properties to understand the influence of the interfacial transition zone [6,21–25]. Due to the limitations of microindentation, such as larger indent comparing with the thickness of ITZ [26], a new method – which has been already used for many other materials [27] – nanoindentation, began to be used in the cementitious composites. Since the cementitious materials exhibit heterogeneous features from the nano-scale to the macroscopic scale, by using the nanoindentation technique, better representation of this heterogeneity at multiple length scales, to ultimately identify the scale where physical chemistry meets mechanics can be obtained [28]. Till now, nanoindentation was widely used to measure elastic modulus and hardness of cement paste cured at different conditions [29–35]. Only few research works were focused on the *studying* of the ITZ between a rigid inclusion and matrix [36–38].

In the present paper, three types of steel fiber reinforced mortars were prepared. Good grinding and polishing procedures were finally determined for the nanoindentation test after several trial procedures had been made. Atomic Force Microscope (AFM) study was carried out to measure the surface roughness. Berkovich indenter was used to investigate the steel fiber–matrix interface and fiber–matrix–aggregate interface differences. Then, the indented areas were studied by Scanning Electron Microscopy (SEM). Influence of the water/binder ratio and silica fume on the characteristics of the profiles of the elastic modulus and hardness of the interfacial transition zones and the corresponding interfacial bond conditions was investigated.

2. Materials and methods

2.1. Materials and proportions of mixes used

The mortar mixes were prepared with cement, sands 0–4 mm and 0–2 mm, water, steel fiber, limestone powder and/or silica fume. Norcem Anlegg cement which is an ordinary Portland cement was used. Silica fume (Elkem Microsilica grade 940-U) in powder form with the content of SiO_2 larger than 90% was used. The limestone powder, used as a filler, has a fineness of 64.3% <30 μm. Sands 0–4 mm and 0–2 mm have fineness modulus of 2.37 and 1.51, respectively. Glenium 151 was incorporated in all mixes as a superplasticizer, having 15% solids content. Straight high carbon steel fiber OL13/.16 has a length of 13 mm and a diameter of 0.16 mm. The detailed particle sizes and densities of all the materials are summarized in Table 1.

Two water/binder ratios of 0.3 and 0.5 were adopted and two different contents of silica fume (0% and 10%) were considered to prepare steel fiber reinforced mortars. The detailed information of those specimens is shown in Table 2, where the numbers in the brackets of columns 4, 5 and 8 indicate the contents of the limestone, Glenium 151 and silica fume as percentages of the weight of cement, respectively. The amount of steel fiber is 0.3% by volume of the mortar. The specimens were identified with numbers designation: the first two numbers indicating water/binder ratio, the second two numbers corresponding to the content of silica fume, the last two numbers corresponding to the content of steel fiber. As an example, mortar 031003 implies a specimen with a water/binder ratio of 0.3, having 10% silica fume and 0.3 vol.% steel fiber.

2.2. Mixing and curing

The steel fiber reinforced mortars were mixed in a flat-bottomed mixer with a maximum volume of 12 l. The mixing procedure was as follows: 1) cementitious materials (including silica fume) and sands were blended for 1 min at lower speed; 2) following the next 4 min, half of the mixing water was firstly added during the mixing, then all the superplasticizer with the remaining water was added; then, the steel fibers were added in small batches to get a good dispersion of fibers in the mixes; 3) stop the mixing, substances sticking in the sides of the mixer were cleaned and mixed into the mixes within 1 min; 4) the mixture was allowed to rest for 5 min, then mixed for an additional 1 min at lower speed.

40×40×160 mm prisms were cast for nanoindentation test. During the casting, steel molds were vibrated in order to evacuate parts of the entrapped air. Then, surfaces of the specimens were carefully smoothed and covered with plastic sheets. Specimens were demolded after 24 h and were cured at 20 °C under water for 28 days.

Table 2
Characteristics of the mortar mixes.

Name	Mix proportion (kg/m^3)							
	Anlegg cement	Free water	Limestone	Glenium 151	Sand 0–4 mm	Sand 0–2 mm	Silica fume	Steel fiber
030003	543.0	153.7	47.2 (8.7%)	10.86 (2.0%)	1399.4	246.9	0.0 (0.0%)	24.2
031003	483.8	149.4	47.4 (9.8%)	12.10 (2.5%)	1399.4	246.9	48.4 (10.0%)	23.4
050003	411.5	201.5	47.3 (11.5%)	4.94 (1.2%)	1399.4	246.9	0.0 (0.0%)	23.4