Cement & Concrete Composites 37 (2013) 276–292

Contents lists available at SciVerse ScienceDirect

Cement & Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

Properties of interfacial transition zones in recycled aggregate concrete tested by nanoindentation

Jianzhuang Xiao [a,*], Wengui Li [a,b], Zhihui Sun [c], David A. Lange [d], Surendra P. Shah [b]

[a] Department of Building Engineering, Tongji University, Shanghai 200092, PR China
[b] Center for Advanced Cement-Based Materials, Northwestern University, Evanston, IL 60208, United States
[c] Department of Civil & Environmental Engineering, University of Louisville, Louisville, KY 40292, United States
[d] Department of Civil & Environmental Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801, United States

ARTICLE INFO

Article history:
Received 6 July 2012
Received in revised form 27 October 2012
Accepted 11 January 2013
Available online 22 January 2013

Keywords:
Recycled Aggregate Concrete (RAC)
Interfacial Transition Zone (ITZ)
Atomic Force Microscope (AFM)
Nanoindentation
Nanomechanical properties
Microstructure

ABSTRACT

The properties of new Interfacial Transition Zone (ITZ) and old ITZ in Recycled Aggregate Concrete (RAC) were investigated by Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM) and nanoindentation. From the SEM images, obvious voids and high concentration of calcium hydroxide can be found in both old ITZ and new ITZ in RAC. Based on the nanoindentation study, it is indicated that the thicknesses of old and new ITZs are in the range 40–50 μm and in the range 55–65 μm, respectively. It is also found that the average indentation modulus of old ITZ is 70–80% of that of old paste matrix, while the average indentation modulus of new ITZ is 80–90% of that of new paste matrix. Additionally, the influences of mix proportion, aggregate types and hydration age on the properties of ITZs in RAC are discussed in this study.

1. Introduction

Recycling of waste concrete is necessary from the viewpoints of environmental preservation and effective utilization of resources. Recycled Aggregate Concrete (RAC) usually either fully or partially uses crushed and sieved waste concrete as its coarse aggregates [1–4]. Researches based on the comparisons of workability, mechanical properties, and shrinkage and creep with respect to conventional concrete have been published to investigate the early and long-term behaviors of RAC [5–10]. However, very limited studies have been reported to investigate the microstructure of RAC and its effect on the mechanical properties [11,12]. As already known, two types of interfaces exist in RAC: the old Interfacial Transition Zones (ITZs) between adhered old cement mortar and natural aggregates, and the new ITZs between new cement mortar and the aggregates (either recycled aggregates or natural aggregates). This is one of the main differences between RAC and conventional concrete. It is widely accepted that the ITZs in RAC have significant influences on the mechanical properties. Ryu found that the failure behaviors of the RAC depended on the relative quality of old ITZ and new ITZ [13]. The strength of the RAC depends on the quality of new ITZ when the quality of old ITZ is better than that of new ITZ. Otherwise, the strength of RAC depends on the quality of old ITZ. To investigate the effect of the recycling process on recycled aggregate, Nagataki et al. concluded that recycling processing level and quality of the waste concrete played a very important role in the characteristics of recycled aggregates [14].

In the past, researchers expressed considerable interests in the micromechanical properties of old and new ITZs in RAC to help understanding the complex mechanical behaviors of RAC such as failure mechanism and durability [5,13–15]. However, due to the difficulties in determining constitutive parameters with the available testing methods, the understanding of the fundamental properties of the ITZs in RAC on submicron or nanoscales is still very limited. With the emergence of nanoindentation technology, it is now possible to directly measure the nanomechanical properties of the thin ITZs [16–18]. The nanoindentation technique is similar to classical indentation, but is capable of producing contact areas and penetration depths on submicron or nanoscales for cementitious materials [19–22]. In nanoindentation tests, the contact area is measured directly from the depth of penetration of the indenter into the specimen surface and the geometry of the indenter [23,24]. Atomic Force Microscopy (AFM) is another increasingly popular tool for studying cement-based materials [25,26], which is often used to assess concrete surface roughness. In order to study the microstructural gradients of the ITZ in concrete, Scanning

* Corresponding author. Tel.: +86 21 65982787; fax: +86 21 65986345.
E-mail address: jzx@tongji.edu.cn (J. Xiao).

0958-9465/$ - see front matter
http://dx.doi.org/10.1016/j.cemconcomp.2013.01.006

Fig. 1. Recycled coarse aggregate (4.75–25.0 mm) (Rossi Contractors Inc. in Chicago, IL).

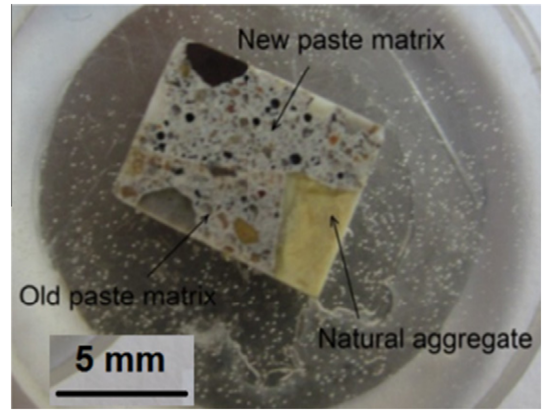

Fig. 2. RAC sample after grinding and polishing.

Electron Microscopy (SEM) is usually applied [27,28]. With SEM technique, one can identify the phase distribution, such as C–S–H or CH, in concrete.

To understand the influence of ITZ properties on RAC properties, the first step is to study how the ITZ properties change when subjected to various influencing factors. Because ITZ is a layer formed between an aggregate and the paste matrix, its properties are affected by both of them. It was found that the cementitious materials grain size distribution, water-to-binder ratio, hydration age, mixing approach, aggregate size, and aggregate type, etc. have effects on the ITZ properties [29–32]. For example, it was found that the thickness of ITZ increases with increasing water-to-binder ratio (w/b) and aggregate size. Moreover, the composition and particle size distribution of the binder can also affect the porosity of the ITZ [33]. Finer particles, such as silica fume, can decrease ITZ porosity and the thickness [34]. Researchers also found that the size and surface texture of the aggregates have great influences on ITZ properties. Compared with gravel, a limestone is more porous and has a rough surface texture. Therefore, the ITZ around a limestone can be relatively stronger and thinner [31]. In order to investigate both the micromechanical properties and phase distributions on micron and submicron scales, this paper utilizes the introduced three advanced techniques (nanoindentation, AFM, and SEM) to study the influences of mix proportion, aggregate type and hydration age on the properties of old and new ITZs mainly including their elastic moduli and thicknesses.

2. Research scope and significance

This research mainly focuses on studying two properties of ITZs: the elastic modulus and the thickness. Knowing the elastic modulus distribution within ITZs can help to improve our knowledge of the failure mechanism and cracking progresses in RAC. And knowing the thickness can help to develop methodologies to improve the quality of RAC by minimizing the ITZ volume.

The objective of this study is to investigate the influences of mix proportion and hydration ages on the characteristics (modulus and thickness) of ITZs in RAC by three testing methods: nanoindentation, AFM, and SEM. This study presents two gaps in the current understanding of RAC behavior by: (1) obtaining a more accurate understanding of local mechanical properties on nanoscales for ITZs in RAC and (2) revealing the differences in nanoscale mechanical properties between old and new ITZs in RAC. The experimental results of this work should be beneficial for future studies to correlate the microstructure and nanomechancial properties to the macroscopic mechanical behaviors of RAC.

3. Materials and methods

3.1. Materials and mixing approach

Recycled aggregates (as shown in Fig. 1) from Rossi Contractors Inc. at the Chicago O'Hare International Airport were used to prepare RACs in this study. The specific gravity and the water absorption capacity of these recycled aggregates were 2.41 and 5.51%, respectively. Most of natural aggregates in the recycled coarse aggregate were limestone, except a few of them were gravel. Due to the high water absorption, the RCAs used for casting RAC specimens were presoaked before mixing. The water amount used to presoak the RCA was calculated according to the water absorption capacity of RCA. The maximum size of coarse aggregates was about 20 mm. Two different RACs were mixed and investigated in this study (Table 1). One was prepared with a water-to-binder ratio (w/b) of 0.42 (noted as RAC I). The Type I Portland cement content was 246 kg/m^{3}and the Class C fly ash content was 61 kg/m^3 in RAC I. The other RAC used in this study had a water-to-cement ratio (w/c) of 0.45, where the Type I Portland cement, water, and river sand were applied (noted as RAC II). Compared to RAC I, no fly ash was used in RAC II mixture. For both mixtures, a Two-Stage Mixing Approach (TSMA) was applied when preparing the RAC specimens [35]. During mixing, the required water was proportionally split into two parts that were added at different timing. All of the samples were cured under 25 °C and 55% relative

Table 1
Mix proportions of RAC I and RAC II.

	w/b (w/c)	kg/m^3					Mixing approach
		Water	Cement	Fly ash	Sand	Recycled coarse aggregate	
RAC I	0.42	129	246	61	854	978	TSMA
RAC II	0.45	200	444	0	702	1054	TSMA

Note: TSMA means two-stage mixing approach.