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Interface characteristics of concrete prepared with recycled aggregates by heating

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Abstract. This study investigates the effects of heating temperature and water-to-binder ratio (W/B) on the interfacial transition zones (ITZ) in recycled concrete, with a focus on multiple interfaces, including the interface between old aggregate and new mortar (LG-XJ), the interface between old aggregate and old mortar (LG-LJ), and the interface between old mortar and new mortar (LJ-XJ). The results show that heating temperature significantly impacts the ITZ microstructure and mechanical properties. Lower temperatures (300°C and 500°C) promote dehydration of old mortar, leading to denser ITZ structures with increased interfacial hardness and reduced transition zone widths. Conversely, at 700°C, decomposition of hydration products increases porosity and microcracking, resulting in decreased interfacial hardness and wider transition zones. Similarly, higher W/B ratios lead to insufficient cement hydration, increased porosity, and looser ITZ microstructures, thereby reducing interfacial microhardness and widening the ITZ. SEM analysis further reveals distinct microstructural characteristics among the interfaces, with the LJ-XJ interface exhibiting better density and uniformity compared to the LG-XJ interface after heat treatment. These findings highlight the importance of optimizing heating temperature and W/B ratio to enhance the interfacial properties and overall performance of recycled concrete.

Keywords: heating temperature; interfacial transition zone; microstructural properties; recycled concrete

1. Introduction

With the acceleration of urbanization, the disposal of construction waste has become an urgent environmental issue. Recycled Aggregate Concrete (RAC) has garnered significant attention as a sustainable building material (Lotfi *et al*. 2015, Bravo *et al*. 2015, Silva *et al*. 2015, Kurbetci *et al*. 2022, Kareem and Hilal 2023). RAC not only mitigates the environmental impact of construction waste but also effectively utilizes limited natural resources. However, during the production process, RAC often introduces certain defects that are particularly pronounced in the interfacial transition zone (ITZ), thereby affecting the overall performance of the concrete (Kou and Poon 2010, Guo *et al*. 2023, Mohamed *et al*. 2022, Mukharjee and Barai 2019).

ITZ is the transitional region between aggregate and cement paste in concrete, and its microstructure and microhardness significantly influence the mechanical properties and durability of concrete (Bru *et al*. 2014, Katz 2004, Tam *et al*. 2007, Al-Bayati *et al*. 2016, Gupta *et al*. 2024). Research has shown that the thickness of the ITZ is typically in the range of a few micrometers to several tens of micrometers, with many incompletely hydrated cement particles, microcracks, and hydration products within it. These microstructural characteristics make the ITZ a weak link in concrete, prone to crack propagation and performance degradation (Leemann *et al*. 2006, Chen *et al*. 2013, Verma and Ashish 2017, Shohana *et al*. 2020). A research team led by Professor Li (Li *et al*. 2017) from Qingdao University of Technology has pointed out that the complex interfacial structure of RAC mainly includes three types of interfaces: the interface between old aggregate and new mortar (LG-XJ), the interface between old aggregate and old mortar (LG-LJ), and the interface between old mortar and new mortar (LJ-XJ). They analyzed the microstructural characteristics of these interfaces from the perspectives of interfacial microhardness and interfacial width. Li *et al*. (2012)and Feng *et al*. (2021) used nanoindentation technology to quantitatively analyze the multiple interfacial transition zones in recycled concrete, revealing differences in interfacial microhardness and pore distribution. Mičušík *et al*. (2012) found that the interfaces of LG-XJ and LJ-XJ are generally more compact than the interface of LG-LJ. Xiao *et al*. (2009) pointed out that the compactness of each interfacial transition zone is closely related to the compactness of the cement mortar. Since the new mortar in recycled concrete is more compact than the old mortar, the structure of the interface between aggregate and new mortar is more compact than that between aggregate and old mortar. This finding provides an important basis for optimizing the interfacial properties of recycled concrete.

Under high-temperature conditions, the interfacial characteristics of concrete undergo significant changes.

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Thermal treatment not only affects the macroscopic mechanical properties of concrete but also alters the microstructure and microhardness of the ITZ. Lu *et al*. (2024) investigated the effects of thermal treatment on the ITZ of RAC and found that heating can improve the interfacial structure, but high temperatures may lead to a decrease in interfacial strength. These changes are mainly reflected in the increase in interfacial porosity and the decomposition of hydration products, which in turn affect the microhardness and overall performance of the interface.

To better understand the interfacial characteristics of RAC under high-temperature conditions, this study systematically explores the effects of thermal treatment on the microhardness and microstructure of the RAC interface. Through experimentation and analysis, this paper aims to reveal the underlying mechanisms of how thermal treatment affects the interfacial properties of RAC, providing a scientific basis for the application of RAC in high-temperature environments.

2. Materials and experiment

2.1 Materials

2.1.1 Cement

Ordinary cement (P.O.42.5) was used for the test. The physical properties of the cement are listed in Table 1.

Table 1 Physical properties of cement

Compression strength (MPa)		Flexural strength (MPa)		Initial condensation final condensation (min)		Specific surface area (m^2/kg)
3d	28d	3d	28d			
18.7	46.3	3.6	7.1	252	301	348

Table 2 Properties of aggregates

Aggregates type	Apparent density (kg/m^3)	Water absorption (%)	Crushing index (%)
Coarse NA	2725	0.46	6.3
Coarse RA	2560	2.32	9.6
Fine NA	2645	1.11	7.4

Table 3 Mix proportions of concrete

No.	Coarse type	W/C	Design of mix proportion (kg/m^3)		
			Cement	Fine NA	SP
A-0	Coarse NA	0.35	450	1300	6.75
A-1	RA-300	0.35	450	1300	6.75
A-2	RA-500	0.35	450	1300	6.75
A-3	RA-700	0.35	450	1300	6.75
B-1	RA-500	0.35	450	1300	6.75
B-2	RA-500	0.45	450	1300	6.75
B-3	RA-500	0.55	450	1300	6.75

2.1.2 Aggregates

The coarse NA with a particle size of 5 mm~25 mm was used. Coarse RA with particle sizes of 5 mm~25 mm were used. Medium sand was used as the fine NA in the test. The main properties of the coarse and fine NA were measured according to the Chinese Standard JGJ52-2006, and the results are shown in Table 2. The coarse RA was heated at constant temperatures of 300℃, 500℃, and 700℃ for 30 min, designated as RA-300, RA-500, and RA-700, respectively, and then placed in the concrete shaking table for 15 min (Su *et al*. 2015). The main properties of coarse RA were measured according to the Chinese Standard GB/T 25177-2010, and the results are listed in Table 2.

2.1.3 Additives

A polycarboxylate superplasticizer (SP) was used as an admixture in a 1.5% cement mass.

2.2 Mix proportions

To investigate the influence of recycled aggregate on interface performance, this experiment employed mortar and distributed pouring of recycled aggregate to fabricate various interfaces required for the study. The specific steps are as follows: First, mortar was poured into a 100 mm × 100 mm × 100 mm cubic concrete mold to a height of half the mold. The mortar was then vibrated to remove air bubbles, and the upper surface of the mortar was leveled. Subsequently, a layer of recycled aggregate was evenly spread on the leveled mortar surface, with half of the recycled aggregate being pressed into the mortar. The mold was then filled with mortar and vibrated to form a complete specimen. To ensure the reliability of the experimental data, three identical specimens were prepared for each group of tests.

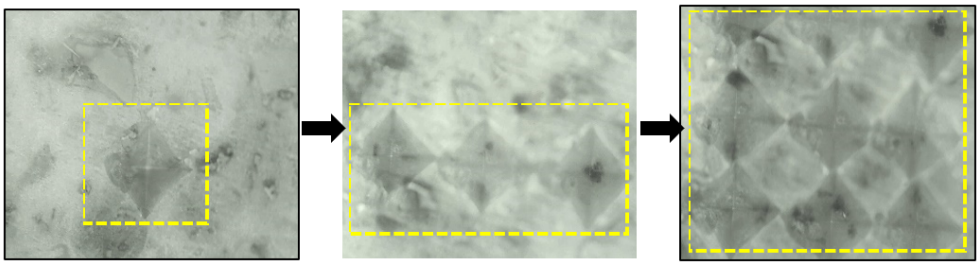
The mortar mix design in the experiment followed the general mix design procedure (JGJ 55-2011). The mix proportions used in the experiment are shown in Table 3.

The specimens were demolded and transferred to a standard curing environment until the specified age. After reaching the designated curing period, the specimens were removed and a thin slice measuring 100 mm × 100 mm × 20 mm was cut from the central part. The slice was then immersed in anhydrous ethanol for 24 hours to terminate the hydration process and subsequently dried in an oven at 60°C until a constant weight was achieved. The side surfaces of the dried slices were selected for grinding and polishing. The surfaces were sequentially ground and polished on a metallographic sample grinder using sandpapers with increasing grit sizes (240CW, 800CW, and P1500). The surfaces were then cleaned with anhydrous ethanol to remove any adhering fine particles. This process resulted in smooth and flat surfaces that met the requirements for subsequent testing.

2.3 Test methods

2.3.1 Microhardness

The HX-1000T microhardness tester was employed to investigate the interfacial characteristics between recycled aggregate and mortar. The testing process is shown in Fig. 1.



(a) Single hit (b) Line dotting (c) Matrix point group

Fig. 1 Interface matrix dot group dotting steps

The microhardness tester was used to quantify the interfacial microhardness and the width of the transition zone between recycled aggregate and cement paste. The magnification was set at 400×, with a load P of 0.05 kg, a holding time of 5 s, and a step length of 10 μm. According to previous studies (Li *et al*. 2017), the indentation patterns for the LG-XJ and LJ-XJ interfaces were rectangular arrays, while the LG-LJ interface used a longitudinal single-line pattern. To minimize testing errors, multiple rectangular arrays were indented near the transition zone to avoid subjective determination of its width.

2.3.2 SEM test

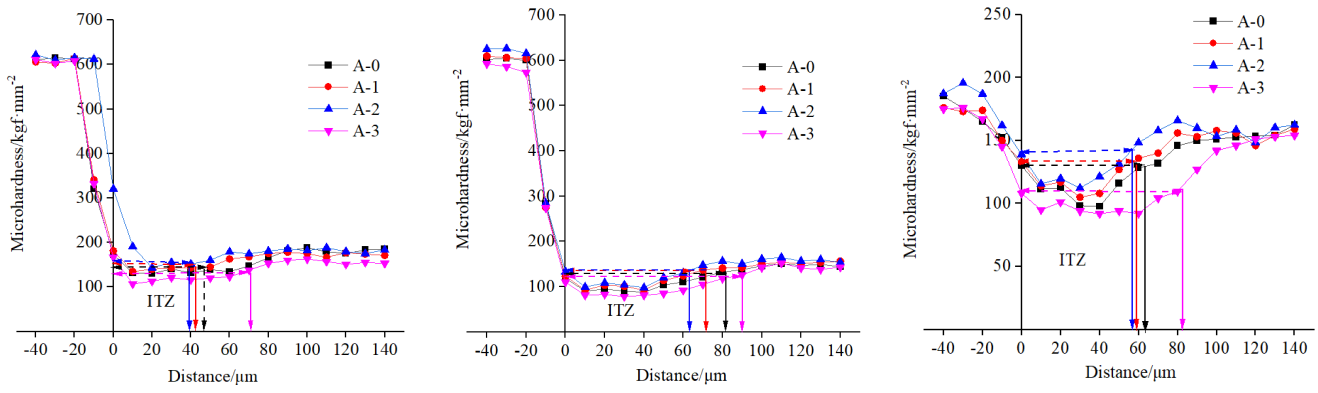
The microstructure of the interfacial region in recycled concrete was observed using a JSM-6390A scanning electron microscope (SEM). Prior to testing, the sample surfaces were coated with gold (Au) to enhance conductivity and imaging quality. The experimental temperature was maintained at 20°C.

3. Results and discussion

3.1 Effect of regenerated aggregate heating treatment on multiple interface properties

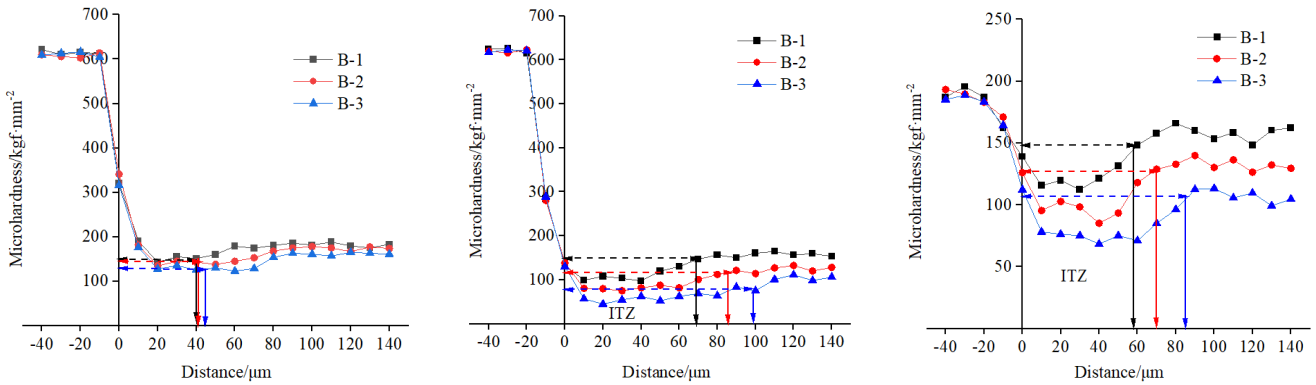
Fig. 2 illustrates the effects of different heating temperatures on the ITZ in recycled concrete. For the interface LG-LJ, heating treatment led to dehydration of the old mortar on the surface of the old aggregate, resulting in a slight increase in interfacial hardness with minimal change in interfacial width. However, when the heating temperature reached 700°C, the interfacial hardness began to decrease as the hydration products in the old mortar decomposed more severely, leading to a significant increase in interfacial width and a looser interfacial structure. For the interfaces between LG-XJ and LJ-XJ, the changes in microhardness and transition zone width followed consistent trends. Specifically, for the LG-XJ interface, the microhardness values ranged from 100.5 to 142.7 kgf/mm² with a transition zone width of approximately 70~75 μm after heating at 300°C. When the treatment temperature increased to 500°C, the microhardness values rose to 102.4~148.3 kgf/mm², and the transition zone width decreased to 65~70 μm. Further increasing the temperature to 700°C resulted in a significant decrease in microhardness values to 67.1~103.4 kgf/mm² and an increase in transition zone width to 90~95 μm. Similarly, for the LJ-XJ interface, the microhardness values were 110.4~143.3 kgf/mm² with a transition zone width of 60~65 μm after heating at 300°C. At 500°C, the microhardness values were 112.3~148.4 kgf/mm², and the transition zone width was 55~60 μm. When heated to 700°C, the microhardness values decreased to 86.4~108.6 kgf/mm², and the transition zone width increased to 85~90 μm.

The underlying reason for these trends is that heating treatment significantly affected the microstructure of the interfacial transition zones. At lower temperatures (e.g., 300°C and 500°C), heating promoted dehydration of the old mortar, reducing the porosity of the transition zone and reorganizing the hydration products, which led to a denser interfacial structure, increased interfacial hardness, and



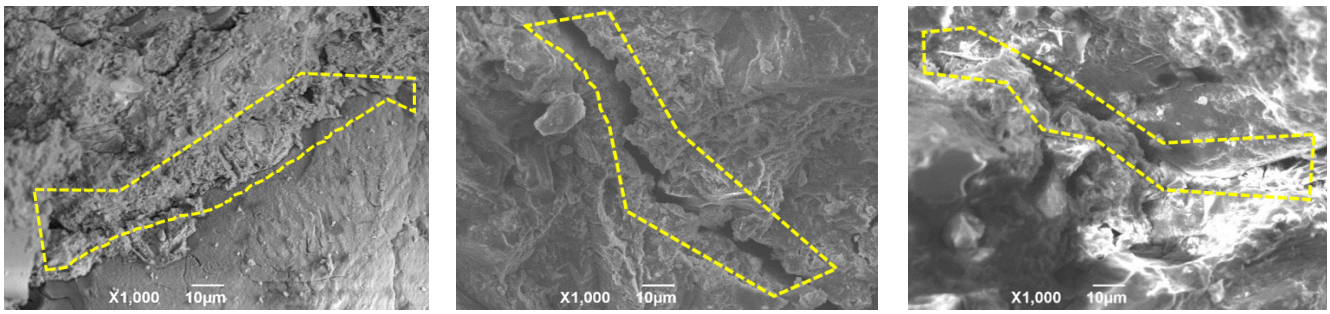
(a) LG-LJ (b) LG-XJ (c) LJ-XJ

Fig. 2 Effect of regenerated aggregate heating treatment on multiple interface properties



(a) LG-LJ (b) LG-XJ (c) LJ-XJ

Fig. 3 Effect of water-binder ratio on multiple interface properties



(a) LG-LJ (b) LG-XJ (c) LJ-XJ

Fig. 4 Multiple interface micromorphology

reduced transition zone width. However, when the temperature reached 700°C, the hydration products in both old and new mortars began to decompose, resulting in microcracking and a significant increase in porosity within the transition zone. This, in turn, caused a decrease in interfacial hardness and an increase in transition zone width. These findings indicate that heating temperature has a crucial impact on the microstructure and mechanical properties of the interfacial transition zones in recycled concrete. Therefore, controlling the heating temperature appropriately is key to optimizing the interfacial properties of recycled concrete.

3.2 Effect of water-binder ratio on multiple interface properties

Fig. 3 illustrates the influence of different water-to-binder ratios (W/B) on the ITZ in recycled concrete. The LG-LJ interface, which had already developed its strength during the service life of the parent concrete, exhibited a relatively stable structure. As a result, the microhardness and width of the LG-LJ showed minimal variation across different W/B ratios, as shown in Fig. 3(a). In contrast, the LG-XJ and LJ-XJ interfaces exhibited consistent trends in microhardness and ITZ width with increasing W/B ratio: the interfacial microhardness decreased, while the ITZ width increased. Specifically, for the LG-XJ interface, the microhardness values ranged from 102.4 to 148.3 kgf/mm² with an ITZ width of approximately 65~70 μm at a W/B ratio of 0.35. When the W/B ratio increased to 0.45, the microhardness values decreased to 90.5~130.4 kgf/mm², and the ITZ width expanded to 80~85 μm. Further increasing the W/B ratio to 0.55 resulted in a further reduction in microhardness values to 57.1~89.6 kgf/mm² and an increase in ITZ width to 95~100 μm. Similarly, for the LJ-XJ interface, the microhardness values were 112.3~148.4 kgf/mm² with an ITZ width of 55~60 μm at a W/B ratio of 0.35. At a W/B ratio of 0.45, the microhardness values decreased to 85.2~122.3 kgf/mm², and the ITZ width expanded to 65~70 μm. When the W/B ratio reached 0.55, the microhardness values further decreased to 66.7~105.2 kgf/mm², and the ITZ width increased to 80~85 μm.

The results indicate that the W/B ratio significantly affects the performance of the LG-XJ and LJ-XJ interfaces in recycled concrete. As the W/B ratio increases, the interfacial microhardness decreases, and the ITZ width increases. This is primarily due to insufficient cement hydration at higher W/B ratios, which leads to increased porosity and a looser microstructure in the ITZ.

3.3 Multiple interface micromorphology

Recycled concrete interfaces with a water-to-cement ratio of 0.35 and subjected to 500°C heat treatment were selected for SEM analysis, and the results are shown in Fig. 4. As can be seen from Fig. 4, there are distinct differences in the widths of the LG-LJ, LG-XJ, and LJ-XJ interfaces.

Specifically, the LG-LJ interface appears relatively dense. This is primarily because this interface was formed during the service life of the parent concrete and has a more stable structure. In comparison, the LJ-XJ interface is denser than the LG-XJ interface, likely because the interface between old mortar and new mortar has undergone reorganization of hydration products after heat treatment, resulting in reduced porosity and a more uniform interfacial structure. Conversely, the LG-XJ interface is relatively wider, indicating that the interface between old aggregate and new mortar still retains some porosity after heat treatment. Although the microstructure of the ITZ has improved, it is not as dense as the LJ-XJ interface. These differences reflect the varying responses of multiple interfaces in recycled concrete to heat treatment, with the LJ-XJ interface exhibiting better microstructural density and the LG-XJ interface showing relatively weaker characteristics.

4. Conclusions

The effect of regenerated aggregate on multiple interfaces after heating treatment were investigated in this study. The conclusions can be drawn based on the experimental results as following:

(1) The heating temperature profoundly affects the interfacial transition zones (ITZ) in recycled concrete. Lower temperatures (300°C and 500°C) induce dehydration of old mortar, resulting in denser ITZ structures with higher hardness and narrower widths. In contrast, at 700°C, decomposition of hydration products increases porosity and microcracking, leading to a looser structure with decreased hardness and wider ITZ. These results underscore the necessity of controlling heating temperature to optimize interfacial properties.

(2) The water-to-binder ratio (W/B) significantly impacts the LG-XJ and LJ-XJ interfaces in recycled concrete. Higher W/B ratios lead to insufficient cement hydration, increased porosity, and a looser ITZ structure, thereby reducing interfacial microhardness and widening the ITZ. These findings highlight the importance of controlling the W/B ratio to enhance interfacial performance.

(3) SEM analysis shows distinct characteristics among the LG-LJ, LG-XJ, and LJ-XJ interfaces after 500°C heat treatment. The LG-LJ interface is relatively dense due to its stable formation in parent concrete. The LJ-XJ interface exhibits better density and uniformity due to reorganized hydration products, while the LG-XJ interface remains more porous and less responsive to treatment. These observations suggest that the LJ-XJ interface is more favorable for microstructural density, whereas the LG-XJ interface requires further optimization.

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