

Study on microstructure of recycled aggregate concrete by scanning electron microscopy: A review

Abstract: Recycled Aggregate Concrete (RAC) is a new type of concrete made by utilizing waste concrete as aggregate after treatment. With the enhancement of environmental awareness and resource conservation, but the microstructural part of recycled aggregate concrete is not as important as the macro performance enhancement aspect by scholars. And Scanning Electron Microscope (SEM) is a powerful tool for microanalysis, which can provide high-resolution images of samples as well as information on surface morphology and microstructure. In this paper, the micro-structure of RAC will be comprehensively analyzed according to the performance characteristics of RAC materials and the relevant analysis technology of SEM. Based on the microscopic technology of SEM, the research progress of the micro-structure of RAC was carried out, and the morphology and distribution characteristics of recycled aggregate particles, pore structure and porosity, the interface combination between recycled aggregate and cement matrix, and the relationship between micro-structure and RAC properties were summarized, which provided theoretical support for the future understanding of the micro-structure of RAC, increased the understanding of the micro-structure of RAC materials, and more systematically understood the application of microscopic analysis methods in RAC.

Keywords: Recycled aggregate concrete; Scanning electron microscope; Microstructure; Durability; Mechanical property

1. INTRODUCTION

RAC is a sustainable building material made from recycled waste concrete or construction waste as aggregate and remixed with fresh cement and admixtures. With the rise of the concept of sustainable development and the increasing pressure on resources and the environment, RAC is receiving more and more attention from the engineering community. However, due to the use of recycled aggregates (RA), the microstructure of RAC is somewhat different from that of conventional concrete. Factors such as binder attachment, calcareous concrete residue, and agglomeration of cementitious materials may exist in recycled aggregates, which can affect the mechanical performance and durability of RAC. Domestic and foreign scholars have conducted relevant studies on the mechanical properties of recycled coarse aggregate concrete: Zaid O et al^[1] found that when recycled aggregate replaces 100% sand, the splitting tensile and flexural strengths are reduced by 14.2% and 14.9%, respectively, and the compressive strength is reduced by 25% compared with that of natural concrete through mechanical property tests. Kazmi et al^[2] investigated mechanical property tests the mechanical properties of coarse fracture behavior and mechanical properties of synthetic fiber reinforced recycled concrete. It was found that the compressive strength of concrete decreased by 23% with the increase of RCA substitution rate, and when the substitution rate of recycled coarse aggregate reached 100%, the concrete splitting tensile strength and flexural strength decreased by 16%

and 23%, respectively. Reddy^[3] discussed the durability of untreated RCA and the corresponding concrete and also explored the improvement in the durability performance of recycled aggregates when treated with appropriate techniques. It was found that the chloride diffusion and capillary absorption parameters of the concrete increased progressively with the increase in RCA substitution and the resistance to carbonation decreased drastically. It was also concluded that immersion of concrete in acetic acid solution followed by accelerated carbonation is a better modification. Bu C^[4] et al. studied the durability of recycled concrete and came to similar conclusions. The recycled aggregate is a waste building material, so the source of recycled aggregate is very different, and the preparation methods are also different (such as crushing method, screening, etc.) will lead to different particle shape and particle size distribution of recycled aggregate. If the pollutants attached to the surface of aggregate (such as oil, old cement paste, etc.) are not effectively removed, it will affect the bonding properties of cement paste, and the reaction characteristics of cement and recycled aggregate may be different, which will affect the generation and distribution of hydration products. For example, the use of wetter recycled aggregates may affect the hydration process of cement and the formation of C-S-H (hydrated calcium silicate) products, which seriously and directly affect the performance of recycled concrete. So it is important to study the microstructure of RAC. The study of its microstructure is of great significance for the in-depth understanding of its performance characteristics and the development of reasonable design, construction and maintenance methods. By observing and analyzing the microstructure of RAC, information on the distribution of RA, particle morphology, pore structure, and interaction with the cement matrix can be understood, thus revealing the relationship and influencing mechanism between the properties of RAC and its microstructure^[5-7].

Among modern testing technologies, SEM has the advantages of large depth of field, high resolution, intuitive imaging, strong three-dimensional sense, wide magnification range, and the sample to be tested can be rotated and tilted in three-dimensional space^[8-11]. It can be used for qualitative and quantitative analysis of elements in micro-areas while studying topography analysis, and is being used as a powerful tool to explore the microscopic material world. It plays a huge role and is widely used in micro-research in biomedicine, physics, chemistry, justice, earth science, materials science and industrial production^[12-17]. The application of scanning electron microscopy (SEM) technology can analyze the structure and chemical composition of concrete from micro and nano scale, provide different morphology, structure and chemical composition, and deepen our understanding of micro-interface^[18]. Scanning electron microscopy combined with energy spectrometer can not only obtain the structure and topography of the sample, but also carry out three-dimensional reconstruction at micro and nano scale, and provide corresponding chemical information and elemental analysis of the sample surface^[19-21]. In recent years, with the development and application of SEM technology, it has become an important tool for studying the microstructure of RAC. SEM technology can provide high-resolution images to help researchers observe and analyze the details of microstructure in RAC, such as particle morphology, pore structure, and interfacial

bonding. In this paper, the microstructure study of RAC based on scanning electron microscope technology is reviewed according to the relevant literature to lay the foundation for promoting the wide application of recycled building materials and realizing the sustainable use of resources and the sustainable development of the environment.

2.PROPERTIES OF RAC

RAC usually refers to new concrete that is formulated using recycled aggregates to partially or completely replace natural aggregates. Recycled aggregates generally come from abandoned buildings and are crushed, cleaned and screened by machines, and contain natural aggregates, old mortar and a combination of the two^[22]. RAC technology can realize the recycling of waste concrete, restore some of its original properties, the formation of new building materials products, thus reducing both the number of landfills and simple piles of waste concrete, but also make the limited resources can be reused, with significant economic, social and environmental benefits.

Recycled aggregates are divided into two main categories, one is RAC aggregate and recycled brick aggregate, we only consider the classification and application of RAC aggregate here: RA is classified into 0-5mm, 5-10mm, 10-20mm, 16-31.5mm specification models, in which most of their use scenarios are substituting or partially substituting for natural sand, used for sand and gravel bedding, road laying , brick making, concrete mixing, roadbed filling, etc. It is suitable for industrial and civil construction projects, municipal engineering, landscaping, decoration and ornamentation, road projects, sponge city construction projects and so on^[23-27].

2.1Mechanical Property

Hao^[28] et al. prepared test blocks with different mix ratios for testing by experimentally using waste bricks to replace natural gravel and waste concrete to replace natural sand. The test results showed that the compressive strength of RAC was related to the water-cement ratio, which had a greater effect on the compressive strength at the age of 3 and 7 days, while the effect on the age of 28 days was not obvious. The compressive strengths of the test blocks with different mix ratios were close to each other, ranging from 21 to 25 MPa.

Xiao^[29] et al. from Tongji University analyzed the effects of the substitution rate of recycled coarse aggregate on the shape of the stress-strain full curve of RAC and the compressive strength, modulus of elasticity, peak and ultimate strain of RAC through the uniaxial compressive stress-strain full curve test of RAC with different substitution rates of recycled coarse aggregate. The study shows that the overall shape of the stress-strain full curve of RAC is similar to that of ordinary concrete, but the stress and strain values at each characteristic point on the curve are different; the ratio of prismatic compressive strength to cubic compressive strength of RAC is higher than that of ordinary mud concrete; the peak strain of RAC is greater than that of ordinary concrete; and the modulus of elasticity of RAC is obviously lower than that of ordinary concrete.

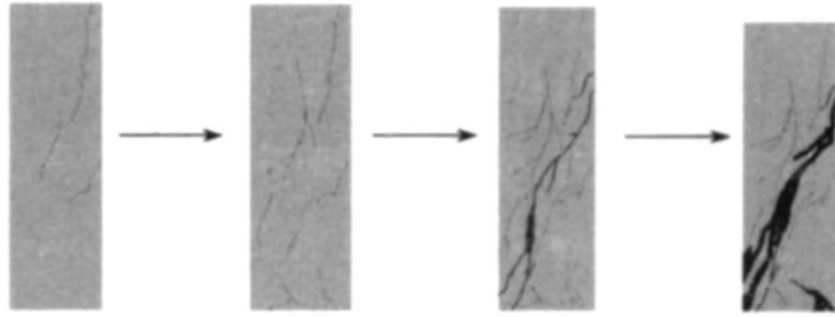


Fig.1 Typical failure process and appearance of recycled concrete prisms

Xu^[30] et al. of Zhejiang University successfully formulated C40-C60 high-performance RAC by using high-quality mineral admixture and high-efficiency water reducing agent, and used electro-hydraulic servo pressure testing machine to carry out uniaxial compression test on the high-performance RAC, measured the stress-strain curve and carried out theoretical analysis, and summarized the mathematical expression for the uniaxial stress-strain curve of the uniaxial compression of the RAC, which matches well with the test results. The mathematical expression of uniaxial stress-strain curve of RAC is summarized, which is in good agreement with the test results.

Wang^[31] collected old test blocks in the laboratory and 23 old bridge components serving in cold areas, prepared recycled concrete specimens, analyzed the influence mechanism of different parameters such as the replacement rate of recycled aggregate, the form of grade, the amount of highly effective water reducing agent and the sand rate on the mechanical properties of recycled concrete, and compared the compressive properties of the old test blocks in the laboratory and the recycled concrete of the old bridge. The results show that increasing the replacement rate of recycled aggregate and sand rate has a negative effect on the compressive strength of recycled concrete. It is suggested to choose continuous gradation when preparing recycled concrete, and pay attention to controlling the replacement rate when applying recycled aggregate in cold area.

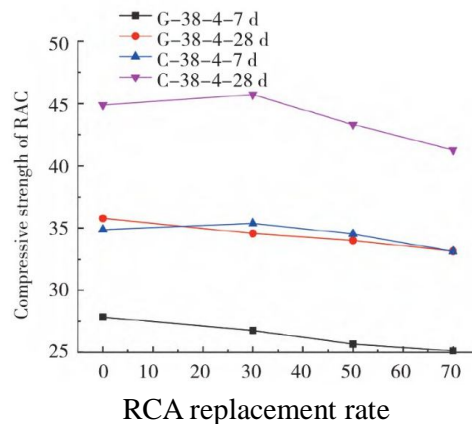


Fig.2 Compressive strength of recycled concrete with different gradation forms
Zhang et al^[32] conducted strength tests on recycled concrete with different

replacement rates in order to study the effects of coarse aggregate replacement rate (0, 30%, 50%, 70%, 100%) and curing age (3, 7, 14, 28d) on the performance development of recycled concrete. The evolution of pore structure in recycled concrete under different conditions was analyzed by using low field nuclear magnetic resonance technique. The results show that the mechanical properties of recycled concrete are the best when the replacement rate is 30%. When the replacement rate exceeds 50%, the mechanical properties of recycled concrete begin to decline significantly. Finally, a strength prediction model is established, which has a good correlation with the test results.

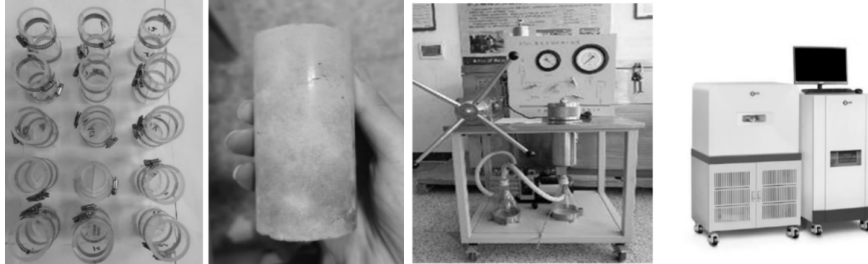


Fig.3 Flow chart of LF-NMR test

2.2 Durability

Based on the results of existing research on the durability of RAC and its influencing mechanism, Zhang et al. explored and analyzed the influencing elements of the durability of RAC from the aspects of aggregates, water-cement ratio, admixtures, supplementary cementitious materials (SCM), and microscopic morphology and summarized the development law of the performance of RAC, and summarized the influences of the mixing amount of recycled aggregates, water-cement ratio, fly ash, and the effect of the admixtures on the seepage resistance of RAC.

Zhu^[33] et al. conducted an experimental study on the permeability properties of RAC by changing the water-cement ratio (W/C of 0.4, 0.5, 0.6) and recycled aggregate admixture (RA of 0, 30%, 60%, 100%), and concluded that the resistance of RAC to chloride ion permeability was better than that of ordinary concrete at low water-cement ratios (W/C of 0.4, 0.5) and lower water-cement ratio could effectively improve the resistance of RAC to chloride ion permeability. It is concluded that at low W/C ratios (W/C of 0.4 and 0.5), the chloride ion permeability of RAC is better than that of ordinary concrete, and that lowering the water-cement ratio can effectively improve the chloride ion permeability of RAC.

Zhang^[34] et al. compared the differences in the permeability properties of natural concrete and RAC under different strengths (C20, C30), water-cement ratios (W/C of 0.42, 0.45, 0.50, 0.55), and sand ratios by means of a concrete permeability test, so as to analyze the factors affecting the permeability properties of RAC and conclude that the permeability of RAC increases with the increase in the strength class of concrete and decreases with the increase of water-cement ratio, and the permeability of concrete increases sharply when the water-cement ratio of concrete is greater than 0.55.

Han^[35] et al. used rapid freeze-thaw method to comparatively study the freezing

resistance of ordinary aggregate concrete (OAC), high-quality recycled coarse aggregate concrete (HRAC), and normal-quality recycled coarse aggregate concrete (NRAC), and at the same time, combined with the rate of mass loss, the relative kinetic elasticity modulus changes to study the quality of RA and the mixing amount of RA on the freezing resistance of RAC under different numbers of freezing and thawing cycles. The test shows that the order of freeze-thaw damage resistance is $OAC > HRAC > NRAC$; when the RA dosage is 100%, the relative dynamic elastic modulus of NRAC is lower than 60%, which does not meet the requirement of F250.

Zhang et al.^[36] discussed and analyzed the factors affecting the durability of recycled concrete from the aspects of aggregate, water-cement ratio, admixtures, auxiliary cementing materials (SCM) and microscopic morphology, and summarized the development rules of its properties, and summarized the research status of the anti-carbonization, anti-freeze-thaw, anti-chloride ion and anti-seepage properties of recycled concrete. It is found that the durability of recycled concrete is worse than that of ordinary concrete, mainly because the addition of recycled aggregate increases the porosity and micro-cracks of the matrix, decreases the compactness and increases the number of interfacial transition zones.

3. INTRODUCTION TO SCANNING ELECTRON MICROSCOPY TECHNOLOGY

3.1 Principles And Applications Of SEM

SEM utilizes a high-energy electron beam interacting with the sample surface to obtain high-resolution images and characterization information. By accelerating the electron beam and focusing it to a very small diameter, a variety of interactions, such as secondary electron emission, reflected electrons, etc., are generated when the electron beam irradiates the surface of RAC. The signals generated by these interactions are received by the detector and converted into images or other forms of data. SEM observation allows detailed characterization and analysis of the microstructure of RAC. The appearance of the SEM is shown in Figure 1.



Fig.4 scanning electron microscope (SEM)

3.2 Progress Of Microstructure Research On RAC

By observing and analyzing the microstructure of RAC, information on the distribution of recycled aggregates, particle morphology, pore structure, and

interaction with the cement matrix can be revealed. This helps to deepen the understanding of the performance characteristics of RAC.

3.2.1 Study On The Morphology And Distribution Characteristics Of Recycled Aggregate Particles

Sun^[37] et al. conducted an in-depth study on the micro-morphological characteristics and elemental composition of recycled aggregates by means of SEM and energy spectrum analysis (EDX) techniques, and analyzed the influence mechanism of recycled aggregates on the performance of concrete. The results show that the morphology of recycled aggregates is diverse and mostly irregular; the surface of recycled aggregates is poorly bonded to the cement paste, which leads to a decrease in the cracking strength; and the fracture interface of the specimens is mainly composed of calcium carbonate and magnesium carbonate. These studies are of great significance for proposing new concrete formulation and control methods, optimizing application techniques, especially new proportion design methods and techniques, and developing new composition systems for concrete. Microscopic picture of sample under SEM is shown in Figure 2.

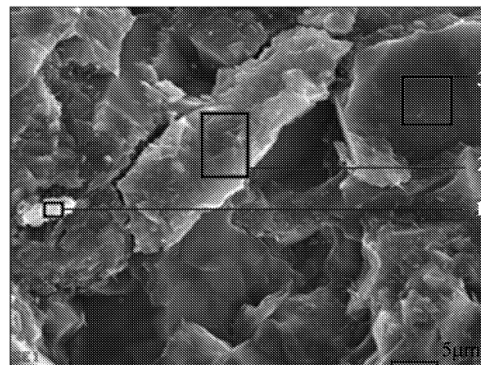


Fig.5 Microscopic picture of sample under SEM

Li^[38] et al. analyzed the flexural compressive strength and micro-morphology of recycled mortar with different particle sizes of recycled aggregate replacing natural mortar step by step by electron microscope scanning technique, and the results showed that: the flexural compressive strength of recycled mortar with recycled aggregate replacing natural sand step by step was larger than that of ordinary mortar, and it increased with the increase of the replacement rate of recycled aggregate; the strength growth of recycled mortar was greatest when the age was 14 d. The strength of recycled mortar was greater than that of ordinary mortar when the natural sand was replaced with recycled aggregate by step by step. The strength of recycled mortar with an age of 14 d increased the most when 100% recycled aggregate replaced natural sand step by step. The picture of morphology of recycled mortar is shown in Figure 3.

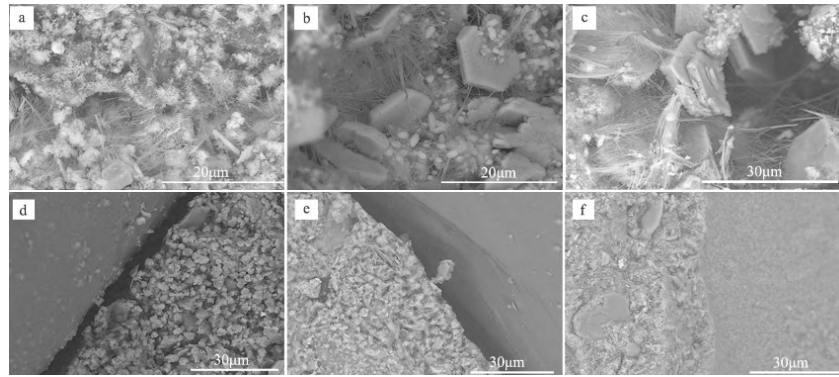


Fig.6 Morphology of recycled mortar

Al-Bayati^[39] et al. improved the physical and morphological properties of coarse RAC aggregates by combining different types of treatments. SEM and energy spectroscopy (ES) analyses were carried out in order to investigate the surface morphology and chemical composition of treated and untreated RAC aggregates. The results showed that the physical properties of RAC aggregates could be maximized by heat treatment at 350°C in combination with short-term mechanical treatment.

3.2.2 Study Of Pore Structure And Porosity

SEM can provide high-resolution images that can reveal the pore structure and pore size distribution of RAC. By observing the type, shape and size of the pore structure, the compactness and pore properties of RAC can be evaluated and the mechanical properties and durability of the material can be further improved.

Li^[40] chose glass fiber to modify the RAC with 25% substitution rate, and took different glass fiber volume rate, special environment type and its action time as variables, and carried out compressive, porosity, impermeability and SEM tests on the RAC with glass fiber after the action of the special environment, so as to analyze and investigate its damage attenuation law at the microscopic and macroscopic levels on the damage to the RAC with glass fiber by the special environment and its damage mechanism. Analyze the damage and damage mechanism of special environment on glass fiber RAC from micro and macro level, and explore its damage attenuation law. The results show that: under the action of special environmental species, the glass fiber volume rate of 1.0% reaches the best, compared with the plain concrete, the compressive strength are improved; porosity is reduced; permeability height is reduced.

Liu^[41] used microscopic tests to study the effect of ceramic sand on the pore structure of cementitious materials, combined with the pore structure parameters measured by microscopic tests, and considered the multi-scale research ideas from microstructure to macroscopic material transport behavior, to study the evolution of the long-term performance of the ceramic powder cementitious materials, and carried out scanning electron microscope tests on the specimens as well as microscopic tests, such as mercury compression. Through the scanning electron microscope test, it was found that the cracks and pores on the surface of the specimen and the transition zone at the cement-mortar interface were gradually filled with hydration products, and the specimen became denser, which led to the improvement of the mechanical and

durability properties of the specimen with the increase of the curing time. The picture of surface micromorphological changes is shown in Figure 4.

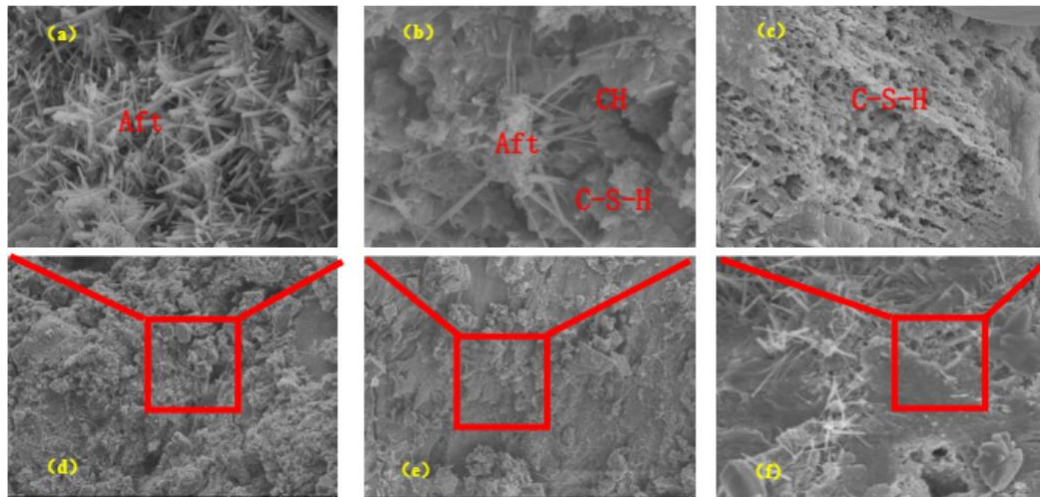


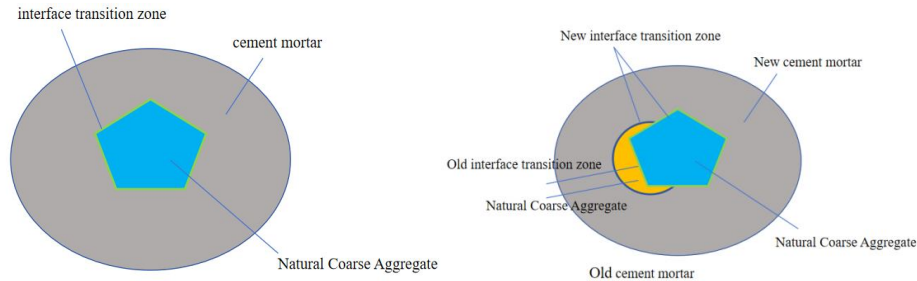
Fig.7 Surface micromorphological changes

Yan^[42] based on SEM-EDS analysis of two-dimensional morphology of recycled aggregate and old mortar, recycled aggregate and new mortar and new and old mortar interfaces and corrosion product evolution within the slurry, studied the structural change rule of multiple interfaces of RAC under the action of internal corrosion as well as the distribution of corrosion products, and revealed the mechanism of the action of sulfate ion-carrying recycled aggregates on the internal corrosion of concrete. It can be seen from the XRD and SEM-EDS test results that the main corrosion products in the mortar are dominated by calcium alumina, and are mainly concentrated in the vicinity of the interface area between the old and new mortar. With the gradual diffusion of ions, a small amount of gypsum is also contained within the mortar when the sulfate ion content is high.

3.2.3 Study Of Interfacial Bonding Between Recycled Aggregates And Cement Matrix

SEM by observing the microscopic morphology and characteristics of the interfacial region of RAC, the bond between the recycled aggregate and the cement matrix can be assessed. This is important for understanding the interfacial properties of RAC, the reinforcement mechanism, and the damage and destruction in the interfacial region.

Compared with ordinary concrete, RAC has a more complex microstructural form, which mainly includes: new cement mortar, natural aggregate in recycled aggregate, old cement mortar carried by recycled aggregate, the interface between old cement mortar and new cement mortar, and the interface between natural aggregate within the recycled aggregate and the old cement mortar, as shown in Figure 1.



(a) Natural concrete microstructure (b) Natural concrete microstructure

Fig.8 Schematic diagram of the internal structure of RAC^[43]

Poon^[44] et al. prepared concrete specimens by using recycled normal strength concrete aggregate, recycled high performance concrete aggregate and natural aggregate as controls and investigated their properties. SEM observations showed that the interfacial region between recycled normal strength concrete aggregate and cement consisted mainly of loose and porous hydrates, whereas the interfacial region between recycled high performance concrete aggregate and cement consisted mainly of dense hydrates. The findings can be explained by the differences in porosity and pore structure of the two types of aggregates and the possible interactions between the aggregates and the cement paste.

Sidorova^[45] et al. in the course of their research suggested that the structure of the cement paste matrix is close to the aggregate and different from the rest of the cement paste. Possible interfacial transition zone (ITZ) formation was compared by nanoindentation tests and SEM, as well as three different properties of aggregate inclusions: limestone, RA, and recycled ceramic aggregate. It is shown that the nature of the aggregate and the W/C conditions determine the microstructural properties of the aggregate-cement paste interface and the mechanical properties of the ITZ.

Rao^[46] et al. discussed the ITZ properties of RAC in terms of hydration compounds, anhydrous cement, and porosity by SEM. Specific steps for SEM specimen preparation were also discussed and the effect of these parameters on concrete strength was further elaborated.

3.2.4 Study On The Relationship Between Microstructure And Properties Of RAC

Wang^[47] et al. modified RAC by the dual modification method of “pre-impregnation mixing and blending”, and tested and analyzed the physical properties of recycled aggregate pre-impregnated with nano-silica sol and the mechanical properties of RAC after the dual modification, and microscopically characterized the modified recycled aggregate concrete by using SEM, EDS, EM and EDS. SEM and EDS were used to characterize the modified recycled aggregate concrete. The EM and EDS characterization showed that the incorporation of nanosilica sol could accelerate the hydration reaction, reduce the Ca/Si, and generate a large number of C-S-H(I)-type gels, so that the new and old cement pastes, as well as the pastes and aggregates are closely linked, and the strength of the recycled aggregate concrete has been significantly improved.

Yue^[48] et al. prepared and tested concrete specimens after immersion in seawater for 0, 30, 60 and 4 months at different replacement rates of 8%, 12% and 16%,

respectively. The aim was to investigate the microstructure and basic properties of recycled aggregate concrete under seawater corrosion and the basic properties of RAC such as compressive strength, modulus of elasticity and chloride penetration depth. SEM was utilized to reveal the microstructure of RA for seawater corrosion. The results show that higher RA content implies higher porosity, lower strength, lower compressive strength, and lower compressive strength and chloride penetration resistance.

Liu^[49] et al. investigated the effects of SSP and metakaolin compounding on the mechanical properties and microstructure of RAC. SEM microtests showed that SSP and MK blending reduced the peak strength of $\text{Ca}(\text{OH})_2$ and generated more additional C-S-H gels as well as increased the percentage of high-density C-S-H in C-S-H, resulting in a significant improvement of the microstructure and interfacial transition zone in the later stages of the RAC, which was attributed to the fact that SSP and MK blending showed better volcanic ash activity and microaggregate filling effect.

Zhang^[50] pointed out that the microscopic properties of concrete can be reflected from the microscopic level. Scanning electron microscopy was used to analyze and summarize the microscopic test results, compare the microstructure of concrete with natural group at 100% substitution rate, analyze its product status and microscopic morphology characteristics, and explore the influence of coarse aggregate substitution rate on secondary recycled concrete. The microstructure contains a large number of holes and cracks corresponding to the analysis of pore structure, which is not conducive to the compressive strength and structural deformation. The observation of the microstructure of recycled concrete confirms the macroscopic measured data of the compressive strength and compressive deformation of the test block and the reason analysis.

4. Conclusion

In a word, the exploration of the microstructure of recycled concrete has an important impact on the control of macro-performance. It was found by scanning electron microscopy that there was an obvious interface between aggregate and new mortar, and the hydration products in the interface were loose and porous particles with irregular shape and a large number of pores. Therefore, the interface bond strength between aggregate and mortar was low. The interface area between the old and new mortar is not obvious, and the two are closely combined. Although there are some pores at the interface, the hydration products at the interface are relatively dense and plate like. Therefore, the interface bonding strength of the old and new mortar is higher. However, some progress has been made in the application of scanning electron microscopy (SEM) in the microstructure of RAC, but there are still some problems and challenges in the following areas: Image interpretation: the interpretation of SEM images requires some expertise and experience in the micro-characterization of RAC. Accurate interpretation and quantitative analysis of complex concrete microstructures, such as cemented regions and phase interfaces, are still challenging.

Disclaimer

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

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