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# Assessing the ITZ microcracking via scanning electron microscope and its effect on the failure behavior of concrete

Tülin Akçaoğlu[a,*], Mustafa Tokyay[b], Tahir Çelik[a]

[a]Civil Engineering Department, Eastern Mediterranean University, Mağusa, Mersin 10, Turkey
[b]Civil Engineering Department, Middle East Technical Eastern Mediterranean University, Ankara 06531, Turkey

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## Abstract

The influence of aggregate size and water-to-cement (w/c) ratio of the matrix on the structure of interfacial transition zone (ITZ) and the interaction between the ITZ and the matrix on the failure process of concrete under uniaxial compression were studied. The ITZ microcracking and the failure process of concrete were investigated experimentally by means of compressive and indirect tensile testing, stress–volumetric strain measurements and microscopic analyses on the model concrete containing single spherical steel aggregate with three different w/c ratios. At low w/c ratios, the rigid and smooth surface texture aggregates made by the ITZ have a significant structural difference compared to the mortar. This was more pronounced for larger aggregates. Higher structural differences between the mortar matrix and ITZ in low w/c ratio composites resulted in accelerated ITZ microcracking at high stress level. The effect of condensed microcracking in a narrower ITZ was reflected in the lower critical stress levels for the low w/c ratio composites with larger aggregates.


*Keywords:* Aggregate; Interfacial transition zone; Tensile properties; Microcracking

## 1. Introduction

Concrete is a highly complex and heterogeneous composite material. The properties of the concrete depend on the properties of its component phases [aggregate, matrix and interfacial transition zone (ITZ) between the aggregates and the matrix] and the interactions between them. ITZ, which is structurally and mechanically different than the matrix, plays a critical role in determining the mechanical properties and failure behavior of concrete composites.

The properties of the aggregates (type, shape, surface conditions, etc.), cement and admixtures and particularly the water-to-cement (w/c) ratio of the mixture are the main factors that form the structure of ITZ and thus its properties [1–8]. The bond strength was found closely related with the type [1,2], shape and surface texture of the aggregate used [3,4,8]. The effect of w/c ratio on the microstructure, thickness and mechanical properties of ITZ was investigated by many researchers [1,6,7,9,10]. It was concluded that by lowering the w/c ratio, the microstructure of the interface could be significantly improved through reduction in porosity. There is also a general agreement that the lower the w/c ratio, the thinner the ITZ [1,6,10] and the greater can be the influence of the type of aggregate [6] on the overall properties of ITZ. Furthermore, for larger aggregates, thickness of the ITZ becomes larger [11]. In researches [1,5,8,12–14] carried out to determine the influence of aggregate, matrix and ITZ properties on the tensile and compressive strengths of concrete, it was found that the interfacial bond was the deciding factor for the tensile strength and played little role on the compressive strength. The influence of the type and the surface texture of the aggregate, on the compressive strength of concrete, however, become paramount as the quality of the mortar is improved.

In connection with the ultimate stress, the significance of investigating the damage process has increased. The fracture of concrete depends on the properties of the particular phases and their interactions. The ITZ properties, has a particular importance on the cracking of concrete. As the w/c ratio increased, the porosity in ITZ is also increased, resulting in the initiation and development of cracks in this zone [9]. The relatively smooth and rounded aggregate provides a weak obstacle to crack initiation and

* Corresponding author. Tel.: +90-542-8807302; fax: +90-392-3651574.
*E-mail address:* tulin.akcaoglu@emu.edu.tr (T. Akçaoğlu).

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propagation [3,4,8]. Besides the effect of the aggregate and ITZ properties, the matrix properties also have a considerable influence on crack initiation and propagation [15,16]. In researches [3,14,17], it was also concluded that, the large difference between the elastic moduli of the matrix and the aggregate induces higher tangential, radial and/or shear stresses at matrix aggregate interface. Critical stress level where rapid and continuous crack propagation starts, is influenced by aggregate properties and elastic compatibility between the matrix and the aggregate [3,14]. Increase in the microcrack concentration at the ITZ while the concrete has been loaded makes the defects act like sources of subsequent macrocrack development. The quality of the matrix is highly dependent on the distribution of these microcracks and their tendency to connect and coalesce. The process from microcracking to macrocracking is thus characterized by the interfacial cracks growing through the matrix and combining with the matrix cracks to form macrocracks. The increasing rate of microcracking into a continuous crack pattern correlates well with the fact that at this critical stress, volume of the concrete no longer decreases but increases due to the pronounced crack formation [14,18].

In summary, it can be concluded that the failure behavior at the ITZ and within the whole material depends on the comparative strengths of the three phases in the composite, i.e., matrix, aggregate and the ITZ between them.

In the present study, the effect of the size of a very rigid, nonporous aggregate with a smooth surface texture, on the formation of ITZ and its impact on the mechanical performance of concrete composites with three different w/c ratios were studied experimentally using indirect as well as direct techniques. The main focus was on the effect of the w/c ratio. For this purpose, single spherical steel aggregates of different sizes were used in mortars of different compressive strengths. This paper is intended to contribute to the understanding of the bond between the matrix and the aggregate for special cases of using very rigid, nonporous and smooth-surfaced aggregates and the subsequent failure process of concrete.

## 2. Experimental procedures

First, three control mortar mixes of 25 MPa (low strength, LS), 36 MPa (medium strength, MS), and 47 MPa (high strength, HS) compressive strength at 28 days were obtained. Then, 100-mm cube specimens were prepared with a single spherical steel aggregate inserted into the center of the specimen right after the moulds were filled. It was verified after split tensile tests that the aggregates were actually at the center of the specimens. The mix proportions and workability of the mortars are given in Table 1. The workability of LS, MS and HS mortars was found to have a flow of 245, 141 and 108 mm, respectively. Diameters of the single aggregate used were 9, 12, 19, 25 and 32 mm.

Table 1
Mix proportions and strength of the control mortar matrices

| Ingredients (kg/m$^3$) | LS | MS | HS |
|---|---|---|---|
| Sand | 1905 | 1905 | 1905 |
| Cement | 525 | 525 | 525 |
| Water | 420 | 320 | 220 |
| Superplasticizer | – | – | 37 |
| 28-day strength (MPa) | | | |
| Compressive | 25 | 36 | 47 |
| Tensile | 1.9 | 2.9 | 3.1 |
| Workability–flow (mm) | 245 | 141 | 108 |

Ordinary Portland cement, natural river sand of 4 mm maximum size, and tap water were used to prepare the mortars. All specimens were cast in 100-mm steel moulds and kept under ambient conditions for 24 h, covered with wet cloth. The moulds were stripped and the specimens were cured under water until testing time.

The microcrack initiation and propagation were analyzed by interpreting the 28-day compressive strength ($\sigma_c$), tensile strength ($\sigma_t$), tensile strength loss (TSL), relation between compressive stress and volumetric strain changes and also scanning electron microscope (SEM) analyses.

Split tensile tests were performed after the application of 40%, 60%, and 80% of $\sigma_c$ in such a way that the plane of tensile fracture was in the direction of the previous compressive loading. For the stress–volumetric strain diagrams, specimens were tested at a constant deformation rate of $1.5 \times 10^{-3}$ mm/s. At the same time, the lateral strains were also recorded. ITZ crack density was obtained by means of a microscopic analysis as described below.

### 2.1. Microscopic (SEM) analyses

In microscopic analyses, low-, medium- and high-strength mortars with 25-mm-diameter steel aggregate inclusion were loaded up to 40%, 60% and 80% of $\sigma_c$. The strained concrete specimens were sectioned transversely and longitudinally using a diamond saw until a small section suitable for SEM analyses were obtained. Just prior to viewing in the SEM, the strained specimens were coated with 200 Å of gold–palladium. It should be noted that each examined cross-section was the surface area of the section in the lateral-casting direction. The microcrack photographs were obtained at three different angles (0°, 45°, 90°) for each surface area that was examined under microscope with a 458× magnification.

Microscopic analyses were attempted to evaluate the relation between ITZ crack density and mortar strength with differing loading levels. Here, the crack density (1/μm), equal to the microcrack length per area unit, is calculated by dividing the total length of the microcracks (μm) that were measured with a ruler per total examined surface area (μm$^2$). All the examined microcrack widths were less than 10 μm and the majority of them had a width of less than 5 μm. The final ITZ crack density, for each specimen tested, was obtained by taking the average of the crack densities measured from the photographs obtained at the three different angles.

## 3. Test results and discussions

The process of microcrack initiation and propagation and the eventual failure of concrete in the single aggregate model can be explained by knowing the intricate relationship between the three phases of concrete and their interaction under loading. The effect of spherical steel aggregate, which is highly stiff and strong with smooth surface texture, plays an important role in the formation of ITZ structure and subsequently the failure process of the composite.

### *3.1. Tensile and compressive strength measurements*

The influence of the presence of a single spherical aggregate on compressive strength ($\sigma_c$) of mortars is shown in Fig. 1. Each test result is an average of at least three specimens. Because of the mismatch in Poisson's ratio, the matrix material above and below the particle was confined, i.e., a state of triaxial compression develops above and below the aggregate particle as described in Ref. [8]. Because the strength of concrete increases under such a triaxial confinement, higher $\sigma_c$ was observed for all composites compared to the control specimens. Fig. 1 also indicates that increasing the aggregate size slightly increases the $\sigma_c$. However, this effect was more pronounced in medium and particularly in high-strength composites due to the relatively better compatibility between the mortar matrix and aggregate phases. This leads to a more efficient transfer of stress between mortar and aggregate.

The effect of the aggregate size and w/c ratio on ITZ strength can be observed in Fig. 2 where tensile strength ($\sigma_t$) measurements are presented. Mortars with an aggregate inclusion had lower $\sigma_t$ and as the aggregate size increase, $\sigma_t$ was further reduced for all composites. The maximum differences between the control specimens and specimens

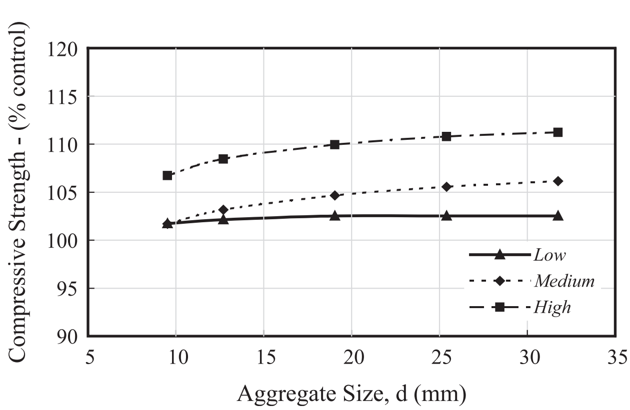


Fig. 1. Effect of aggregate size on compressive strength of LSC, MSC and HSC.

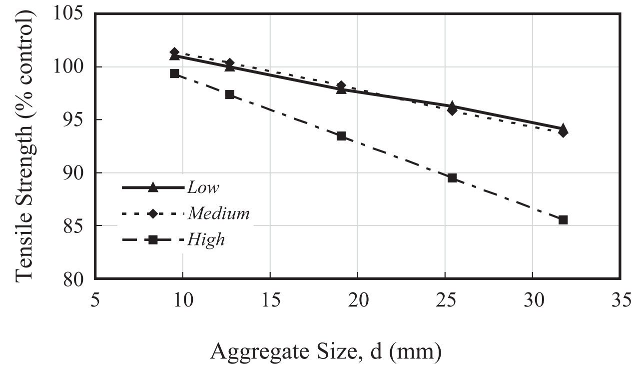


Fig. 2. Effect of aggregate size on tensile strength of LSC, MSC and HSC.

containing aggregates were about 6% for both low-strength composites (LSC) and medium-strength composites (MSC) and 14% for high-strength composites (HSC). It was also noted that the rate of reduction in $\sigma_t$ with increasing aggregate size was higher in HSC. Thus, it can be concluded that ITZ bond strength deteriorated more compared to the matrix strength for low w/c ratio composites for all aggregate inclusions. This was because of the formation of higher initial tensile strains in HSC that made its ITZ critical due to being structurally more different than its matrix in comparison with the other two composites. In other words, the additional tensile strains, which were introduced due to the aggregate inclusion, were higher in HSC and resulted in a higher $\sigma_t$ reduction. The observed decrease in the $\sigma_t$ with increasing aggregate size in all types of composites was due to an increased stress concentration and microcracks in the vicinity of the aggregate with increased surface area.

Another important parameter of ITZ is its thickness and the concentration of microcracks and voids relative to its thickness. Because of the lower w/c ratio, the ITZ is expected to be narrower [1,6,10]. This leads to more condensed stress concentrations and microcracks in the narrower ITZ of HSC. Thus, for larger steel aggregate inclusions (which are rigid, nonporous and smooth) and lower w/c ratios, ITZ becomes more critical.

### *3.2. Crack development*

#### *3.2.1. ITZ microcracking*

The experimental work performed through microscopic analysis revealed the compressive stress-permanent deformation in the ITZ phase of concrete after unloading the specimen. The results can be used to better understand and quantify the general relationship between stress levels (40%, 60% and 80% of $\sigma_c$) and crack development in the ITZ of three different w/c ratio composites. Fig. 3a presents the measured crack density in the ITZ region as a function of w/c ratio of the composite for three different loading stages. The occurred damage in the ITZ decreased almost linearly with decreasing w/c ratio of the mortar matrices up to 40% of $\sigma_c$ loading. However, for 60% of $\sigma_c$ loading, this

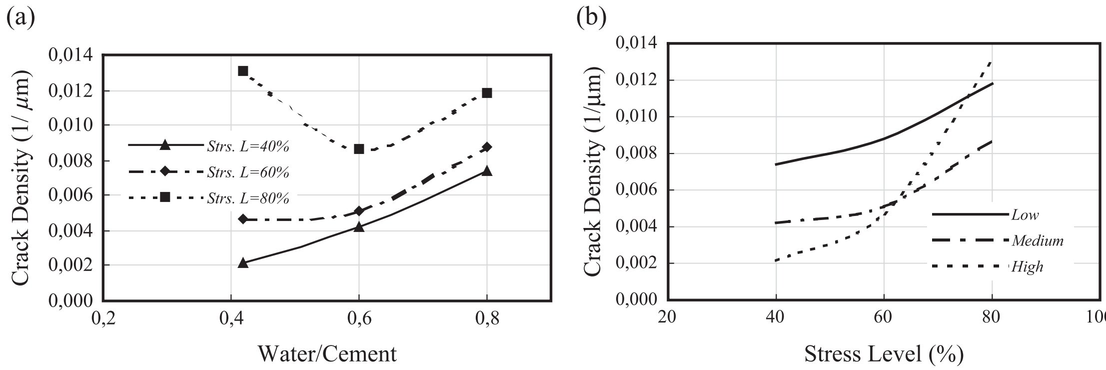


Fig. 3. ITZ Crack density as a function of (a) w/c ratio for three loading levels, (b) loading level for three types of composites.

linearity in the occurred damage was lost. While the highest damage still occurred in LSC, the damage in MSC and HSC were approximately equal. With 80% of $\sigma_c$ loading, damage observed in the ITZ of HSC exceeded slightly that of LSC and it was lowest in the MSC. Fig. 3b is an alternate presentation of the crack density as a function of stress level for the three composite types studied. The accelerated increase in the crack density, particularly in HSC beyond 60% of $\sigma_c$ loading is more evident.

There are two obvious factors affecting the microcracking process in the ITZ. The first is the relative strength of the ITZ with respect to the mortar and the second is the strength of the mortar itself. The bigger the difference between the strengths of the ITZ and the mortar, the higher is the tendency of microcracking in the ITZ. In addition, a stronger mortar matrix forces the cracks to be confined within the ITZ. The nonlinear microcracking in HSC and its acceleration above 60% of $\sigma_c$ loading can be explained by the two factors described above. For the case of LSC, an appreciable amount of cracking occurred in the matrix as well, due to its comparatively low matrix and ITZ quality than the other two composites. Thus, an almost linear increase in the microcrack density was observed with increasing load. The occurred damage in the ITZ for MSC was also nonlinear. Microcracking in MSC was higher than HSC for a loading level less than about 60% of $\sigma_c$ and lowest for higher loading levels. The lower rate of increase in microcracking of MSC with increased load compared to HSC was because the ITZ and mortar strengths were closer to each other in MSC in comparison with HSC. On the other hand, microcracking in MSC was away from being linear as in LSC because it had comparatively much higher matrix quality than LSC.

### 3.2.2. TSL measurements

The loss of tensile strength, in response to an applied compressive stress, is referred to as 'damage' [19]. Microcracking was observed directly by an appreciable loss of tensile resistance in the direction normal to the microcracks. Fig. 4 presents the relationship between damage and aggregate size for LSC, MSC and HSC after applying 40%, 60%, and 80% of $\sigma_c$. The extent of damage was highest in LSC and lowest in HSC at all compressive stress levels applied. For a specific compressive stress, as the aggregate size

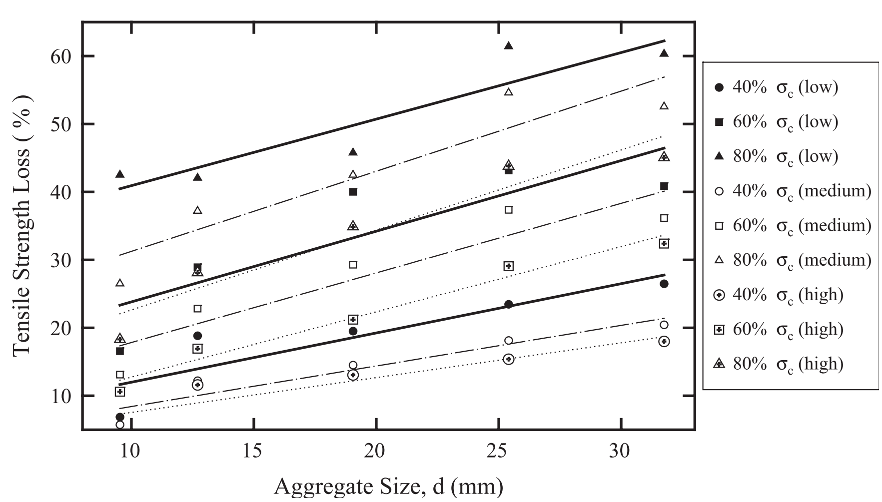


Fig. 4. Effect of aggregate size on TSL in LSC, MSC and HSC.

increased, TSL also increased. This was an indication of the importance of ITZ in damage process for all types of composites. Maximum TSLs of about 60%, 55% and 45% were observed for low-, medium- and high-strength mortars, respectively, at 80% of the compressive strength loading. This regular decrease in the TSL can be attributed to decrease in the w/c ratio, in other words, to the matrix quality. The observed lowest damage in HSC was because the decreased w/c ratio led to a matrix with reduced amount of pores and cracks, thereby resulting in higher fracture resistance. Increased quality of mortar matrix surrounding the aggregate confined the cracks and forced them to accumulate mostly in the ITZ until continuous crack propagation started.

### 3.2.3. Stress strain measurements

Volumetric strains obtained from longitudinal and lateral strains were investigated to determine the effect of aggregate size on the critical stress ($\sigma_{cr}$) at which rapid and continuous crack propagation starts. Cracks initiate at the interface depending on its strength and the amount of stress present in that zone. With increased load, initially present cracks either in the ITZ or in the matrix start to grow in length, width and number. The propagation of internal cracks is uniform but random up to a certain stress level. With further increase of the load, ITZ cracks start to propagate through the matrix. The additional stress required for the concrete composites to reach the $\sigma_{cr}$ level depends on the amount of created lateral stresses along with the material properties (e.g., w/c ratio, aggregate size) of individual constituents.

The $\sigma_{cr}$ levels for three aggregate sizes are shown in Fig. 5. This figure indicates the variation in $\sigma_{cr}$ level for three w/c ratios. The $\sigma_{cr}$ levels for LSC (w/c = 0.80) were just below the ultimate stress (around 95% of ultimate) and did not show any noticeable difference with increasing aggregate size. For MSC (w/c = 0.60) $\sigma_{cr}$ level was slightly lower than LSC. In contrast, for HSC, the $\sigma_{cr}$ level was noticeably lower and decreased with increasing aggregate size from approximately 82% of $\sigma_c$ to 75% of $\sigma_c$ for 19- and 32-mm aggregates, respectively.

With reduced w/c (0.42) ratio, a more homogeneous and brittle matrix was achieved. This resulted in a lower ability of redistributing the stress and lower energy absorption capacity. For this reason, less damage was observed in HSC in TSL measurements presented in Section 3.2.2 for all loading levels, although the microcracking at the ITZ of HSC varied significantly beyond 60% of $\sigma_c$ loading. The accelerated microcrack accumulation within the narrower ITZ of HSC beyond 60% of $\sigma_c$ loading presented in Section 3.2.1, together with a less extensive microcrack pattern of the matrix contributed to a comparatively sudden failure resulting with a lower critical stress. This shows the critical role played by the ITZ on the critical stress level. Another factor contributing to the sudden failure of low w/c ratio composites is the presence of a very rigid and smooth surface aggregate which increases the flow of the matrix around the aggregate resulting in higher lateral tensile stresses.

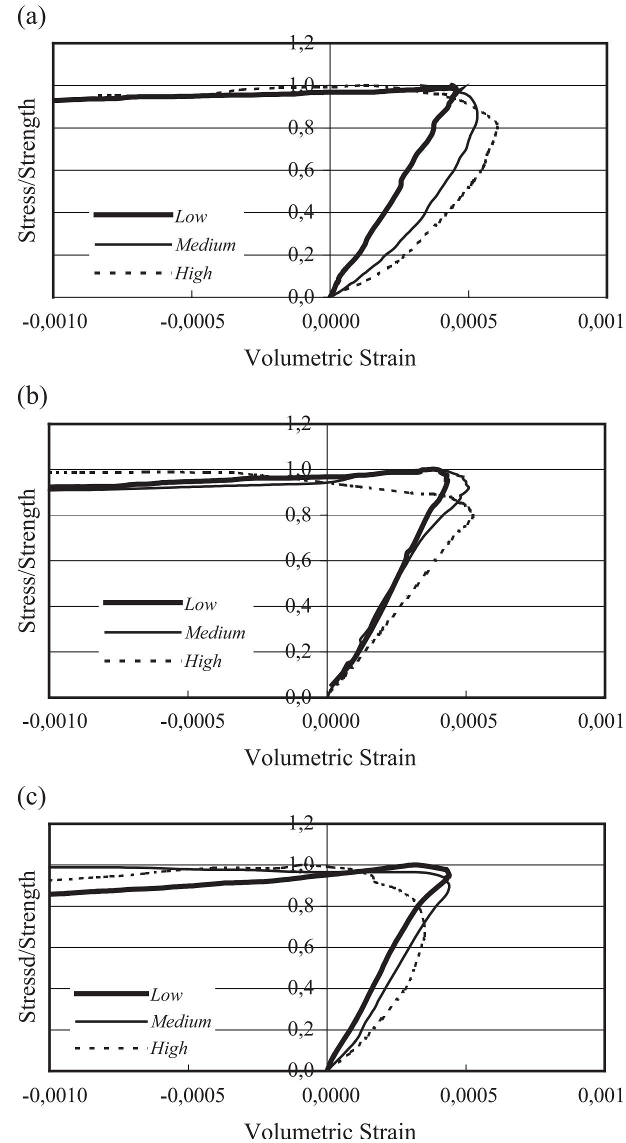


Fig. 5. Stress/strength–volumetric strain curves of LSC, MSC and HSC produced with (a) 19 mm aggregate size, (b) 25 mm aggregate size and (c) 32 mm aggregate size.

## 4. Conclusions

1. With larger aggregates, lower w/c ratio matrices resulted in more critical ITZs with a large difference in strength compared to the matrix. This was promoted by the adverse effect of the rigid and smooth aggregate with increased matrix quality.
2. The interfacial bond was observed to be the deciding factor for the tensile strength and played little role on the compressive strength. The tensile strength decreased as

the aggregate size increased. The rate of tensile strength reduction with increasing single aggregate size was higher in HSC.

3. At low stress levels, ITZ microcracking increased linearly with increasing w/c ratio, which can be attributed to the initial weakness of ITZ. However, at higher stress levels, the mortar strength, its strength difference with the ITZ, and the ITZ thickness became influential. Microcracking tended to be highly nonlinear for HSC.
4. The critical stress for rapid crack propagation for LSC was close to the ultimate stress and did not show any noticeable difference with increasing aggregate size. For MSC, it was slightly lower than LSC. In contrast, for HSC, the critical stress was noticeably lower and decreased with increasing aggregate size.
5. With reduced w/c ratio, less overall damage was observed up to 80% of $\sigma_c$ loading level. Although the microcracking in the ITZ of HSC increased significantly beyond 60% of $\sigma_c$ loading, the overall damage in HSC was still the lowest. The accelerated microcrack accumulation within the narrower ITZ of HSC beyond 60% of $\sigma_c$ loading, together with a less extensive microcrack pattern of the matrix, contributed to a comparatively sudden failure resulting with a lower critical stress. This shows the critical role played by the ITZ on the critical stress level.

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