

Usability of microstructural investigations of concrete-like composites; aggressive factors monitoring

Filip CHYLIŃSKI¹ , Lech CZARNECKI¹ , and Thomas MATHIA²

¹ Instytut Techniki Budowlanej, 00-611 Warsaw, Poland

² Laboratoire de Tribologie et Dynamique des Systèmes, École Centrale de Lyon, 69134 Écully, France

Abstract. The microstructure of a material is a key factor in determining its properties and durability. This paper highlights key findings from microstructural investigations of concrete-like composites, with a focus on their implementation potential. It introduces a usability function and explores trends in its classification to demonstrate the potential of various microscopic techniques in addressing research questions within the field of building materials. This segment of the analysis focuses on various aggressive factors that affect the microstructures and might cause corrosion of concrete-like composites. The findings underscore that the impact of corrosive agents on Portland cement composites cannot be comprehensively assessed without a detailed investigation of their microstructure, emphasizing the pivotal role of microscopic techniques in the evaluation of concrete-based materials. Furthermore, significant advancements are anticipated in the near future as image analysis becomes increasingly supported by artificial intelligence.

Keywords: microstructure; concrete-like composites; SEM; concrete corrosion; usability function.

1. INTRODUCTION

This study aims to investigate how microstructural examinations of building materials contribute to understanding the mechanisms influencing their overall performance and long-term durability. Furthermore, the article examines the utility and effectiveness of microscopic methods within the domain of building materials science. As concerns the building material, it is not enough to say that the material fulfills the technical requirements of the given application at the time of testing. We need to ensure that it will also meet those requirements in the future; in all service life of the building, usually over 50 years. There is no other engineering field where the life cycle of engineering work can be measured by the multiple of its creator's lifespan. The distance to the microstructure damage state in the case of building materials is equivalent to the time separating us from a disaster. This is an extremely complicated issue requiring a responsible approach. At the engineering level, for instance, more than 30 factors can be mentioned which affect the durability of concrete [1]. This assessment is based on interim tests, accelerated aging tests, past experiences, and engineering intuition. Microstructural investigations appear to be a promising scientific tool for making such durability predictions [2–4].

Not only do advancements in microscopic techniques enhance the usability of these methods in assessing the current condi-

tion of a material but also facilitate the prediction of its future state. Central to these advancements is the ability to observe microstructures – material structures visible through microscopy. This article focuses on external aggressive factors affecting materials and the role of microscopy in understanding and mitigating these effects. The study aims to demonstrate the importance of microstructural investigations of building materials and their value in gaining knowledge on several processes that influence the macro performance and durability of building materials. It also tries to define the function of usability of microscopic techniques in the area of building materials.

2. ENGINEERING OF DEFECT INSPECTION

Different types of microscopic techniques in microstructural investigations of building materials defined the usability function (Fig. 1).

The shape of the postulated function of microstructural investigation usability (MIU function) was prepared by analyzing the field of building materials (Fig. 1). The first stage of a linear progression of the curve is related to the increasing resolution of the optical microscopy. In this technique, increasing the resolution within this range does not require significant effort in sample preparation. The shape of the MIU function at another stage, which is related to electron microscopy, varies from the first stage. The usability of increased resolution does not improve significantly, as it is associated with greater challenges in sample preparation compared to optical microscopy. However, the ability to further increase the resolution helped overcome diffi-

*e-mail: f.chylinski@itb.pl

Manuscript submitted 2025-01-30, revised 2025-04-23, initially accepted for publication 2025-05-01, published in August 2025.

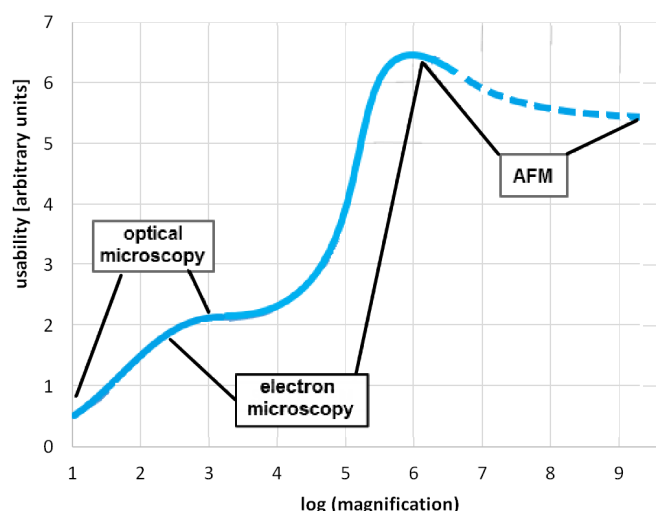


Fig. 1. Diagram of microscopic visualization in building materials engineering based on half a century of expertise

culties and rapidly increase the MIU function value. Reaching the highest available resolutions for electron microscopy techniques leads to further difficulties and limitations, particularly in sample preparation. The third range of resolution in the MIU function was determined using atomic force microscopy (AFM). There is a decrease in usability related to difficulties with sample preparation and long-lasting scans during observations, but also the limitations of cases for which high resolutions are used. It must be noted that, in areas other than building materials, the shape of the MIU function might be different.

Metrology is the science of measurement. Types of metrology can be classified depending upon the quantity under consideration, such as the metrology of length and angles, but also force, displacement, time, and acceleration, for example. Therefore, various suitable measurement units are used. The result of the scanning microscopy is just the microstructural picture – unique in the sense of details and accuracy. A routine way is restricted to the measurement of length and angles – quantities that are expressed in linear or angular terms. However, the micro- or even the nano-scale of observation presents a compelling opportunity to investigate a more fundamental attribute, like durability – or more precisely proximity to disaster. Could we predict collapses before they happen? Could we define a tipping point of ongoing phase transition? An affirmative answer to these questions would mean a breakthrough in this field. Yet, this remains a challenge for the future. In this paper, we attempt to establish the current state-of-the-art and evaluate the likelihood of discovering how to measure the “proximity to a disaster”. What kind of units we should use in such a case? As materials become more complex, meaning they are more multi-component and multiphase, then the metrological aspect becomes more complicated. Thus, the need for more subtle measurement methods arises, making the measurement process itself a subject of consideration, as illustrated by the examples provided in Section 4.

There are non-destructive/semi-destructive tests. It makes a significant difference to be able to state that a structural ele-

ment was in good condition but is now unfit for use after testing, or, conversely, to confirm through non-destructive or semi-destructive testing that the element remains and will continue to be reliable [5–7].

Concerning the above, a few questions arise.

Images obtained using various microscopic techniques might be key in discovering the essence of the investigated phenomenon, especially when they are conducted at the elemental level. However, some techniques might be more efficient than others. This prompts a question: Could the MIU function be a useful instrument for the metrological assessment of microscopic methods?

Regarding composite materials, the phenomenon of synergy takes place. This means that the composite features are not simply the sum of the properties of its individual components. Synergy, as the effect of interaction between components, could either positively enhance durability or devastate the microstructure. What types of microstructures promote positive and negative synergy in composite concrete-like materials exposed to internal aggressive factors?

Is there a possible way of assessing the microstructure of material in the scope of potential failure of the investigated construction element?

The creation of new types of composite materials leads to the formation of more complicated thermodynamic systems. These systems cannot be fully assessed across the entire range of possible constituent proportions before being implemented, which creates a certain area of risk of unforeseen failures for the end product. There arises a question: Can building material microstructure assessment provide the opportunity to identify semi-stable states before the material is placed in construction?

3. STRUCTURAL LEVELS IN BUILDING MATERIALS ENGINEERING

According to the level of magnification at which the observations are conducted, applications of microstructural research in building materials engineering can be classified as macro-, micro-, and nanoscale (Fig. 2) [8, 9].

At the macroscale, macroscopic defects in materials, such as lack of adhesion or cracks, might be observed. Microscale is suitable for observing microcracks and some larger defects in the microstructure, such as the crystallization of expansive phases or the effects of leaching some constituents from the material matrix. The nanoscale is the level of molecules and atoms at which, for example, the essence of adhesion of different materials, or lack thereof, might be studied. In addition, the nature of concrete-like binders and the causes of eventual defects should be investigated. Microscopic methods allow us also to indirectly determine the porosity and pore size distribution in the cement matrix. Analysis of elemental composition in micro areas facilitates the assessment of the Si/Ca and Al/Ca ratios, which provide insight into the internal structure of the C-S-H phase [10, 11].

The effectiveness of microscopic investigations is largely attributed to their relative ease of application. Although mi-

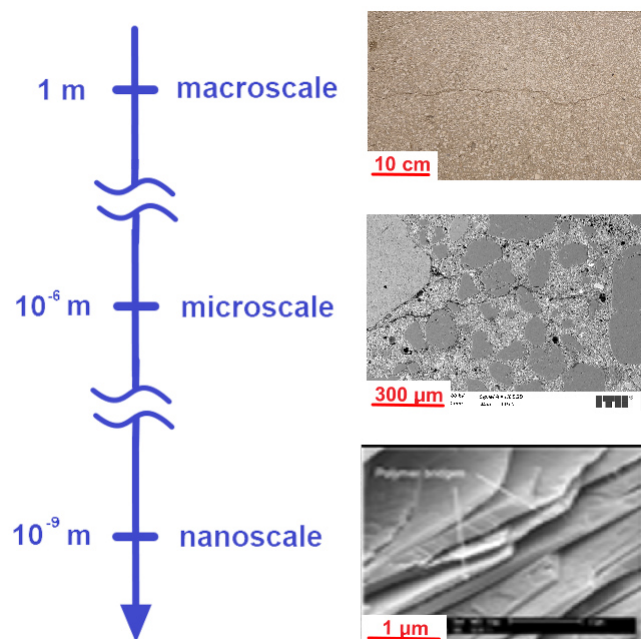


Fig. 2. Structural level in building materials engineering ([8], updated)

crostructural analysis is applicable across various fields, this study concentrates on building materials, with a specific focus on concrete.

Tests of building materials mainly focus on their physio-mechanical properties and on their durability to ensure they meet the expected service life. The microscopic methods might be useful in developing new material compositions with better mechanical properties and durability as well as in predicting how the addition of new constituents or changes in the manufacturing process might influence their properties and durability. In addition, microscopic investigations help to understand why some additions work better than others and how to modify them in the expected way. Changes in the microstructure of materials, particularly concrete-like materials, result in changes in their technical properties. Microstructural changes in hardened concrete are mainly caused by the influence of the surrounding environment and its aggression. However, some changes might be caused by the inner factors related to the maturation of concrete, as well as by the activation of certain aggressive constituents built within [12]. Figure 3 presents the main areas where microstructural examination of concrete, such as building materials, might be useful.

Analyzing the usability of microstructural investigations of concrete-like building materials three main sections are proposed (Fig. 3):

- Effects of aggressive factors on the microstructure of concrete
 - Influence of additional waste materials on the microstructure of concrete
 - Non-Portland cement concretes used in building engineering
- This paper focuses on the first section: “Aggressive action”.

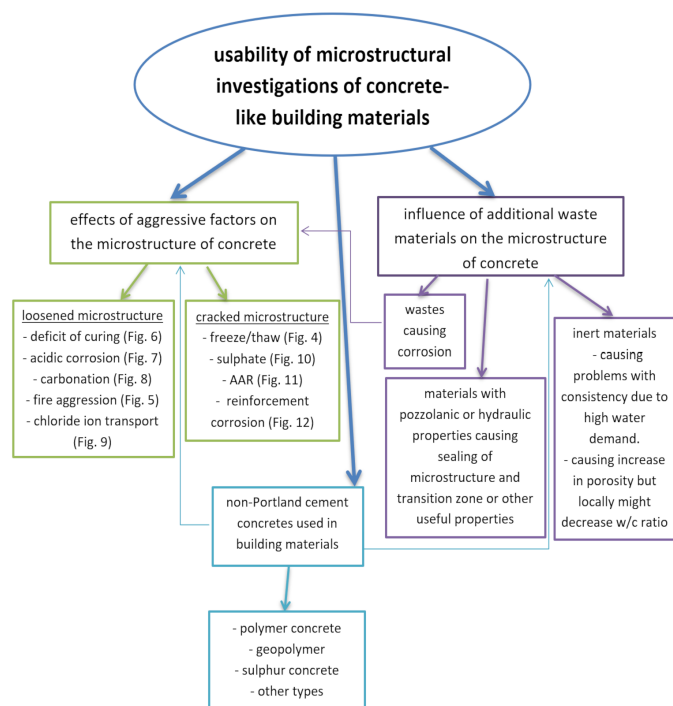


Fig. 3. Usability of microstructural investigations of concrete-like building materials

4. IDENTIFICATION OF AGGRESSIVE ACTIONS ON THE MICROSTRUCTURE OF CONCRETE

4.1. Leading mechanisms

There are two intrinsic corrosion mechanisms observed as effects of external aggressive actions:

- Physical mechanism – phase transition of water – freeze/thaw (Fig. 4), evaporation caused by heat exposure (Fig. 5)
- Chemical reaction mechanism conducted with a physical process involving products of reaction

The reaction products are non-adhesive and soluble in water and might be washed out (Figs. 6–9). The reaction products have a greater volume than the substrates, which causes inner tension resulting in cracks (Figs. 10–12).

4.2. Physical mechanism

Some physical processes might cause degradation of the microstructure of cement composites by expansive phase transformation. The main example of this process is the freezing and thawing of water in a porous concrete matrix. Also, water evaporation caused by heating during exposition to fire might be corrosive. This process might be considered a physical one as long as the temperature does not reach the decomposition point of some constituents and only involves the release of water from the microstructure. In most cases, the first chemical decomposition (of Portlandite) occurs at temperatures of about 460°C. Therefore, the cement composites should be chemically stable even up to ca. 400°C.

Aggression caused by freeze/thaw

Concrete is a porous material. When the temperature of the hardened concrete drops below 0°C , the water filling the pores freezes. The temperature of this phase transformation depends on the concentration of various ions present in the water solution, as well as on the diameter of the pores [13]. The smaller the pore diameter, the lower the freezing temperature. In gel pores with the lowest diameter (less than a few nanometres), water may never freeze in a natural environment [14]. Saturated gel pore water can freeze from -30°C to -80°C [15]. The difference in the freezing point between the pores is the initial factor of concrete frost damage. The phase transformation of water from liquid to solid state is conducted with an increase in volume of approximately 9%, which leads to a high tension in the microstructure of concrete. When several freeze/thaw cycles occur, they might affect the formation of microcracks and, eventually lead to the destruction of concrete [13, 16]. Microscopic microstructural investigations might be extremely useful for diagnosing the degradation of concrete exposed to low temperatures. Cracks in the microstructure are easily visible even at the first stage, when their impact on the compressive strength is not so obvious. Examples of microstructures in the areas near the surface of concrete exposed to freeze/thaw aggression are presented in Fig. 4.

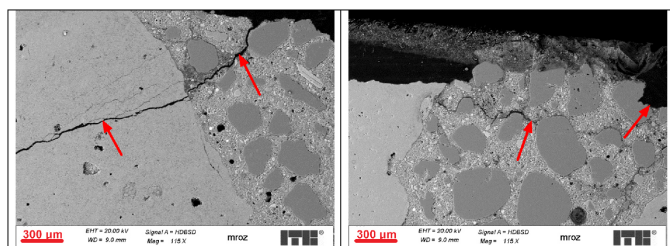


Fig. 4. Effects of freeze/thaw on the microstructure of concrete (arrows mark degraded areas)

Studies on the influence of curing temperature on the pore size distribution in concrete and its further durability in freeze/thaw aggression environments show that samples cured at temperatures ranging from -5°C to standard curing conditions had compressive strength comparable to those of the reference samples, as well as similar numbers of small capillary pores [17]. However, curing concrete at temperatures below -10°C caused an increase in the porosity of concrete, mostly in the range of macropores, which caused a decrease in the compressive strength. Microscopic analysis combined with mercury intrusion porosimetry (MIP) contributed to reaching these conclusions.

Freeze/thaw aggression and microstructure degradation may also occur in environments where de-icing salts are used. The mechanism of this type of microstructural degradation is more complicated than the one previously discussed. A review of recent progress in the theory of the mechanism of this type of aggression shows that the analyzed mechanisms incorporated water phase transition and chloride and sulphate damage [18]. Freeze/thaw microstructure damage models help to understand

how a wide spectrum of factors might influence the corrosiveness of this type of aggressive environment. The main role in this mechanism is the water phase transition; however, damage to concrete is accelerated by the crystallization of de-icing salts in the pore structure, which might cause excessive tension depending on its composition and concentration. Most of the microcracks that formed during the freeze/thaw cycles ran parallel to the surface of the tested specimen, which is the cause of the typically observed scaling effect in this type of corrosion. Microscopic examination of the microstructure is necessary for a complete and thorough analysis of this type of corrosion mechanism.

Fire aggression of cement composites

The exposure of concrete to high temperatures caused by fire might be highly corrosive. The effect of such corrosion depends mainly on three factors: the temperature, time of exposure, and composition of concrete. Some other factors might be important such as initial humidity, heating rate, porosity, sample size, and shape. Analyzing changes in concrete properties and its microstructure after being heated up to 800°C , it was discovered that corrosion caused by high temperatures could be divided into three stages: $20\text{--}200^{\circ}\text{C}$, $200\text{--}300^{\circ}\text{C}$ and above 300°C . In the first stage, there were observed compressive strength losses caused by increasing porosity and releasing water bound in the hydrated phases, which is a physical process. In the second stage, the rising temperature did not strongly affect the compressive strength. Increasing the temperature to values higher than 300°C caused a monotonic decrease in strength [19]. SEM investigations showed that, at this temperature, the rehydration of cement particles continued to generate new Portlandite and C-S-H phases. Increasing the temperature up to 800°C resulted in a growing relative proportion of capillary pores with diameters above 200 nm in the total porosity. The effects of fire exposure on the mechanical properties of fiber-cement boards were studied, and it was discovered that in the temperature range of $300\text{--}400^{\circ}\text{C}$, the bending strength decreased rapidly [20, 21]. To find an explanation for this observation, the material microstructure was examined. Microstructural analysis revealed that within this temperature range, the cellulose fibers used in the production of fiber-cement boards were lost due to high temperatures caused by fire. This loss of fibers is a key reason for the rapid changes in mechanical properties. Examples of such changes in the microstructure of concrete exposed to fire are shown in Fig. 5.

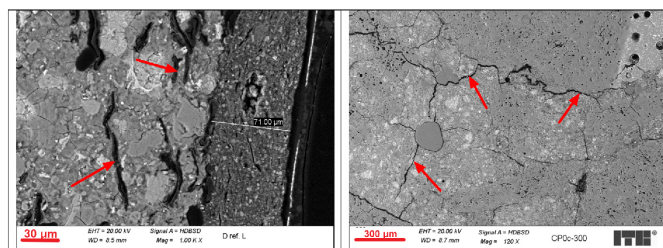


Fig. 5. Effects of fire aggression on the microstructure of concrete (arrows mark cracks in microstructure) [20]

Using microscopic methods and their ability to access the porosity, changes of such property might also be a subject of analysis in a function of thermal load.

4.3. Chemical reaction mechanism and physical process

4.3.1. Chemical reaction with leachable products

The leaching mechanism is initiated by the reaction of an aggressive environment causing leaching of the cement matrix, mainly Portlandite, starting from the surface of the concrete. Further aggression leads to corrosion of the deeper parts of the composite, causes loss of the microstructure, and separates the aggregate from the cement matrix. In general, this process can be divided into two steps – first, a chemical reaction that generates soluble products, and second, the dissolution of these products in water and subsequent leaching thereof from the cement matrix.

Deficit of curing of concrete

Curing fresh concrete is a crucial process, and neglecting it can lead to a decrease in the concrete mechanical properties and durability [22–24]. The effects of its progression might be observed in the microstructure of the near-surface areas of concrete. Chyliński *et al.* [25] analyzed the changes in the microstructure of uncured and cured concrete using various methods and several curing compounds. They found that the tensile strength in the surface areas of the concrete tested using the pull-off method varied significantly. Investigations of the microstructure helped determine the causes of the observed changes in the mechanical properties of concrete. Allowing the cement matrix to mature with an insufficient amount of water leads to the formation of a more porous microstructure and an incomplete C-S-H phase. Moreover, a relatively weak transition zone between the cement matrix and aggregates is also caused by the lower amount of Portlandite due to the reduced level of clinker hydration (Fig. 6).

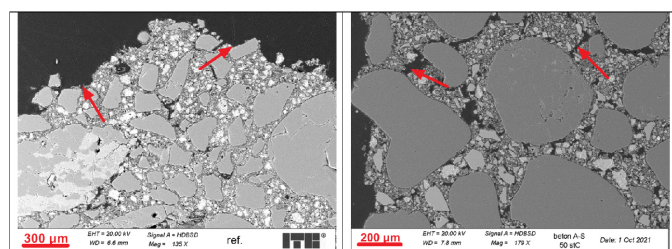


Fig. 6. Effects of deficit in curing on the microstructure of concrete (arrows mark degraded areas)

The analysis of the impact of rapid curing methods including steam, electric, and microwave curing on the properties and microstructure of concrete shows that those methods led to efficient early strength growth. However, it was observed that rapid curing might lead to late shrinkage, causing the formation of microcracks and increased porosity which might further affect durability [26]. Microstructural investigations were useful in the assessment of different types of curing compounds and

rapid curing methods as well as in determining their influence on the porosity and expected durability of concrete.

Acidic corrosion of Portland cement matrix

Acidic corrosion is caused by aggressive acidic solutions that react mainly with the constituents of the cement matrix, although some aggregates, such as calcites and dolomites, might also corrode in acidic environments. The mechanism of corrosion of the cement matrix generally starts from the reaction with Portlandite and the leaching of the products of the reaction, which causes loss of the microstructure. Investigation of the effects of acidic corrosion on the microstructure of Portland cement mortar and alkali-activated fly ash-slag composite using sulphuric acid and acetic acid proves that the leaching mechanism differed for each type of acid [27]. The corrosion caused by acetic acid caused greater damage than that caused by sulphuric acid. Microscopic investigations helped understand the mechanism of corrosion and explain the differences. The changes were caused mainly by the formation of calcium sulphate (gypsum) as a product of the reaction of sulphuric acid with calcium from Portlandite and the C-S-H phase. The formation of gypsum affects the sealing of the microstructure, inhibiting further corrosion. However, this process was not observed in an aggressive environment with acetic acid because the solubility of calcium acetate is much greater than that of gypsum. The aggression of acetic acid solution on concrete and its influence on its microstructure was studied in [28]. The influence of the water-cement ratio on the damage to the microstructure of concrete caused by the aggression of acetic acid as a simulation of corrosion in agricultural concrete tanks was studied. Microstructural investigations helped explain why concrete with a lower water-cement ratio is more durable in such an environment. Microanalysis performed using EDX shows that exposure to an acetic environment results in the leaching of calcium ions from the C-S-H phase near the surface. This process weakens the microstructure, leading to the release of aggregates due to the deterioration of the transition zone (Fig. 7).

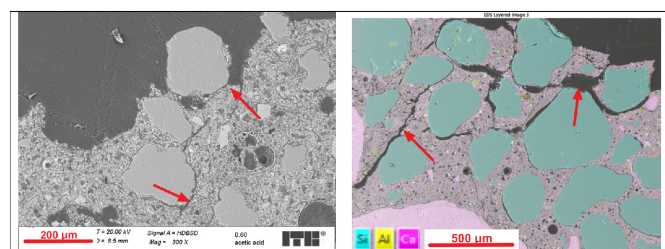


Fig. 7. Effects of acidic aggression on the microstructure of concrete (arrows mark degraded areas)

Carbonation of Portland cement matrix

Carbonation is an effect of the aggression of carbon dioxide on concrete or other Portland cement composites. Carbonation occurs when carbon dioxide reacts with Portlandite and forms calcium carbonate in the first stage. This reaction causes partial sealing of the microstructure and slows down further processing.

However, when concrete is in contact with water, particularly soluble carbon dioxide, it becomes more aggressive by forming calcium hydrogen carbonate which is soluble in water and initiates the leaching process. Carbonation of Portlandite causes a decrease in the pH value, which might induce corrosion of the reinforcement in the presence of Portlandite in the cement matrix [29]. Microstructural investigations may also be useful in this regard. Examples of the microstructures in the areas near the surface of concrete exposed to carbonation are shown in Fig. 8.

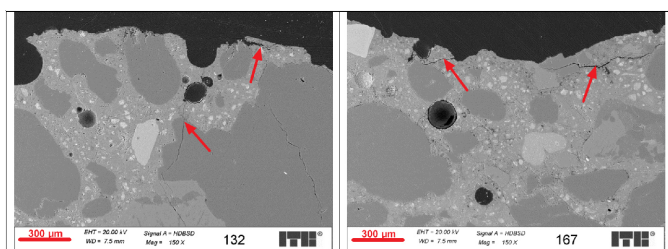


Fig. 8. Effects of carbonation on the microstructure of concrete (arrows mark degraded areas)

The analysis of the impact the addition of different types of fly ash has on the depth of carbonation of concrete led to a discovery that such an addition, particularly of calcareous fly ash, might deepen carbonation [30]. Microstructural investigations helped elucidate this phenomenon. The addition of fly ash, which is an active material, affects the sealing of the microstructure and prevents further penetration of carbon dioxide. However, using fly ash as a part of cement causes a decrease in the concentration of Portlandite in the cement matrix which is a source of hydroxide ions, causing an increase in the pH value.

Reaction of chloride ions with Portlandite

Chloride ions can cause leaching corrosion by forming soluble chlorides. It is best manifested by the formation of calcium chloride as a result of the reaction of chloride ions with Portlandite. The leaching effects observed during microstructural examinations are very similar to acidic aggressions [28,31]. Examples of microstructures in the areas near the surface of concrete exposed to chloride ion aggression are shown in Fig. 9.

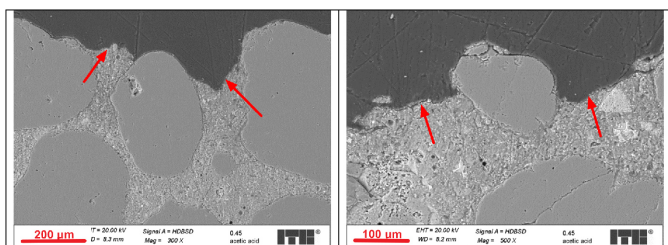


Fig. 9. Effects of chloride ion transport on the microstructure of concrete (arrows mark degraded areas)

Chloride ions easily penetrate the porous microstructure of concrete due to its relatively high diffusion coefficient [32–34]. Preventing this type of aggression concentrates mainly on sealing the microstructure of concrete or adding a sealing layer on

its surface. Material modifications result from the addition of active materials with pozzolanic properties or hydraulic binders other than Portland cement, e.g., slag. Active additives might also help reduce the amount of Portland cement, which causes a decrease in the concentration of Portlandite in concrete and often increases the durability of concrete in chloride environments [35–37]. Some inert materials may also help prevent chloride aggression; however, their role is limited by their grain sizes. To effectively seal the microstructure, micro fillers must contain very small particles, preferably at the nanoscale. This requirement introduces new technical challenges, as these particles can form unnecessary agglomerates, making even distribution within the cement matrix difficult. Moreover, most of them cause problems with the rheology of the concrete mix due to their high water demand [38–40]. There are also some special additives, such as graphene which, in relatively small amounts, help decrease the corrosive effect of chloride ions [37, 41]. In all these cases, microstructural investigations might be useful in observing the effectiveness of sealing the microstructure in preventing chloride aggression and in explaining the mechanism of corrosion in the examined concrete, which provides insight into how to improve the material.

4.3.2. Chemical reaction with expansive products

There are several types of aggression causing inner expansion as a result of chemical reactions, where products have a higher volume than the substrates. In this paper examples of corrosive chemical processes which form products with excessive volume sulphate aggression, alkali-aggregate aggression, and corrosion of steel reinforcement have been discussed. The mechanism of this type of corrosion might be divided into two stages. In the first stage, the reaction within the inner parts of the cement matrix yield products of a greater volume than the original substrates. In the second stage, the products generate inner tension within the microstructure. As a main result of this type of corrosion, cracks in the microstructure of cement composites are observed.

Sulphate corrosion of Portland cement composites

Sulphate corrosion of concrete is mostly related to the exposure of concrete to marine environments. However, aggressive sulphate ions may also occur in groundwater, sewage, or industrial wastewater [42]. The sulphate corrosion mechanism can be divided into two phases [43]. In the first phase, sulphate ions react with Portlandite which affects the formation of gypsum. Gypsum has a larger volume than Portlandite; however, in most cases, it causes sealing of the concrete microstructure without excessive tension that might cause microcracks. The second phase involves the subsequent reaction of the gypsum formed with calcium aluminate which causes the formation of secondary ettringite. The volume of the formed ettringite is much larger than that of the substrates of this reaction, which results in high tension in the microstructure and causes microcracks, macrocracks, and destruction of concrete [44, 45]. Examples of the microstructures of concrete exposed to sulphate aggression are shown in Fig. 10.

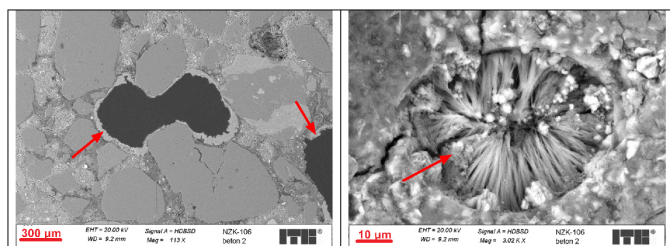


Fig. 10. Effects of sulphate aggression on the microstructure of concrete (arrows mark degraded areas)

Examination of the microstructure of concrete containing different types of cement and with various water-cement ratios which were immersed in water containing sulphate ions revealed that there were two types of sulphate solutions: one with magnesium sulphate and the other with sodium sulphate [46]. By analyzing the microstructure of corroded concretes, it was concluded that the formation of secondary ettringite is usually conducted with the formation of thaumasite. Cracks in the microstructure created as an effect of the formation of ettringite allow carbon dioxide to enter deeper into the microstructure, which increases the formation of thaumasite. It was also discovered that the type of cation related to sulphate plays an important role. Comparing magnesium and sodium ions, the magnesium ions reacted in the near-surface areas with hydroxide ions to form magnesium hydroxide and magnesium carbonates which partially sealed the microstructure and limited the diffusion of aggressive factors deeper into the microstructure. The observed depth of sulphate corrosion was greater in concrete immersed in sodium sulphate than in magnesium sulphate. Studies on the sulphate corrosion mechanism and developing methods for preventing would not be possible without a microscopic investigation of the microstructure.

Corrosion of Portland cement composites caused by alkali-aggregate reaction

Alkali aggregate corrosion occurs when concrete contains alkali reactive aggregate and some other circumstances promote this reaction such as appropriately high alkali concentration, humidity or water saturation, and temperature. There are two main types of alkali aggregate reactions: the alkali-silicon reaction (ASR) and the alkali-carbonate reaction (ACR). What links these two types of inner aggression is that during the process reactive aggregate grains react with alkalis from the cement matrix and form products of much higher volume than the substrates, causing tension which in turn could result in the formation of cracks in the microstructure. During the ASR reaction, the expansive product was silicone gel, and in ACR, it was mainly magnesium hydroxide [47]. When investigating damaged concrete structures where cracks occur, microscopic methods are indispensable for identifying the cause of cracking as an ASR or ACR through the analysis of the microstructure and products of the reaction. Sometimes, cracks might not occur in such a spectacular way, especially when fine grains of aggregates are reactive. In this case, silicon gel is leached on the surface of the cement composite (Fig. 11).

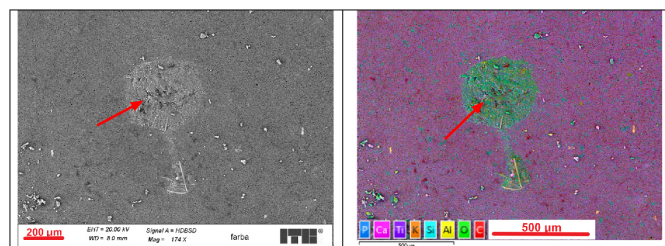


Fig. 11. Effects of alkali-aggregate reaction on the microstructure of concrete (arrows mark degraded areas)

Examination of the influence of de-icing salts containing alkalis and wet/dry cycles on initiating and accelerating ASR using microstructural observations involving SEM-EDX revealed that wet/dry cycles with exposure to NaCl solutions promoted ASR [48].

Damage to Portland cement composites caused by reinforcement corrosion

Concrete mostly contains reinforcement in the form of bars or fibres which increases the bending strength of the composite. Steel reinforcement is the most commonly used reinforcement method. High pH values in concrete and reinforcement covers of appropriate thickness ensure adequate durability of construction by creating a passive layer on the surface of the steel elements. However, when the pH value of the reinforcement cover decreases, for example, during the corrosion process, the corrosion of steel begins. The steel corrosion process is well known and described in the literature [49]. The essence of this process and one of the most destructive stages is that the corrosion products of steel have a larger volume than the substrates, which generates a large pressure between the steel element and reinforcement cover. This leads to detachment of the cover which exposes the steel reinforcement and accelerates its corrosion. Examples of the microstructure of the steel reinforcement surface received from the corroded concrete are presented in Fig. 12.

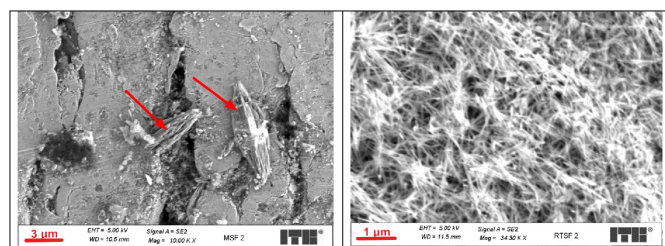


Fig. 12. Surface of steel reinforcement received from corroded concrete (arrows mark products of corrosion)

In the examination of corrosion-affected reinforced concrete microstructure, focusing on the transition zone between the steel and the cement matrix, microstructural analysis helped conclude that the steel-concrete interface plays a crucial role in the chloride threshold limit, and air voids in the transition zone cause earlier corrosion [50]. The low water-cement ratio of cement paste affects the sealing of the microstructure, particularly the

transition zone, which prevents the initiation of corrosion, even with a relatively high concentration of chloride ions in the environment. They also investigated the influence of the alumina cement paste coating of steel bars on protection from chloride aggression. Microstructural analysis shows that the conversion of alumina cement increases the porosity of the transition zone which accelerates the corrosion process in marine environments. Furthermore, methods for corrosion improvement of carbon steel concrete reinforcement by modifying the steel microstructure were studied [51]. Three types of low-carbon steel with different grain sizes were used. The concrete samples with reinforcements were immersed in water and water with NaCl. The use of microstructural examinations helped discover that steel with fine grains passivated faster than the other. However, the passive layer protects steel with coarse grains against the chloride solution over a longer period.

5. CONCLUSIONS

The durability and other technical characteristics of building materials are cast in their microstructures. Microscopic methods appear to be an effective means for advancing knowledge and comprehension in this domain. Their high resolution and wide range of magnification make them a particularly versatile and practical tool for analyzing building materials. These methods are especially valuable for identifying the mechanisms behind observed processes, particularly in concrete-like composites. This study demonstrates that the effects of aggressive factors causing corrosion in Portland cement composites cannot be fully observed or understood without examining their microstructure, emphasizing the importance of microscopic techniques in studying concrete-based materials.

The MIU function defined in this paper is not complete since only the shape of a curve is given and the axis of ordinates, i.e., the Y-axis, is neither characterized by values nor accompanied by an equation. However, analysis of the usability of microstructural investigations serves as a valuable tool for the evaluation of microscopic methods. While there remains a need for uncertainty assessment, an initial approach appears promising. However, significant progress is expected because image analysis will be supported with artificial intelligence. Exploring the microstructure at higher magnifications offers significant benefits, but it also presents challenges. As magnification increases, the analyzed area of the composite becomes exceedingly small, making it difficult and uncertain to draw conclusions about the entire element.

Consequently, scientists often repeat the same analyses across various regions of the composite to confirm or refute earlier observations. Several tools help collect dozens of images from the selected area. However, an analysis of each of them belongs to scholars. To increase the potential of microscopic methods the automatization of the image analysis process needs to be conducted. While there are tools or software available for statistical analysis of an image, they are far from what AI might bring shortly, for example, in object identification or determination of the type and mechanism of corrosion.

The examples of microstructure in various Portland concrete scenarios presented in this paper demonstrate that microstructure is not merely a source of visually appealing images but serves as a critical diagnostic element. It functions as an engineering tool that enables the categorization of the primary mechanisms driving material degradation. With regard to the questions posed at the beginning of this paper, microscopical examinations might become a diagnostic non-destructive or semi-destructive tool for materials microstructure capable of predicting collapses before they happen and also of defining a tipping point in ongoing phase transitions.

REFERENCES

- [1] L. Czarnecki and D. Van Gemert, "Scientific basis and rules of thumb in civil engineering: Conflict or harmony?," *Bull. Pol. Acad. Sci. Tech. Sci.*, vol. 64, no. 4, pp. 665–673, 2016, doi: [10.1515/bpasts-2016-0076](https://doi.org/10.1515/bpasts-2016-0076).
- [2] D.H. Murcia and M.R. Taha, "Emerging Materials and Technologies for Next-Generation Sustainable and Resilient Polymer Concrete," 2025, pp. 47–58.
- [3] M. Reda and M. Elshikh, Mohamed Yousri Dawood, "Selection of Sustainable Construction Method Using Analytical Hierarchy Process," *MEJ-Mansoura Eng. J.*, vol. 42, no. 2, pp. 1–9, 2021.
- [4] M.T. Reda Taha, Mahmoud Bassuoni, "Nanotechnology for Improved Concrete Performance," *Spec. Publ.*, vol. 335, 2019.
- [5] K. Schabowicz, "Non-Destructive Testing of Materials in Civil Engineering," *Materials (Basel)*, vol. 12, no. 19, p. 3237, Oct. 2019, doi: [10.3390/ma12193237](https://doi.org/10.3390/ma12193237).
- [6] J. Hoła, J. Bień, Ł. Sadowski, and K. Schabowicz, "Non-destructive and semi-destructive diagnostics of concrete structures in assessment of their durability," *Bull. Pol. Acad. Sci. Tech. Sci.*, vol. 63, no. 1, pp. 87–96, Mar. 2015, doi: [10.1515/bpasts-2015-0010](https://doi.org/10.1515/bpasts-2015-0010).
- [7] J. Hola, L. Sadowski, and K. Schabowicz, "Nondestructive identification of delaminations in concrete floor toppings with acoustic methods," *Autom. Constr.*, vol. 20, no. 7, pp. 799–807, Nov. 2011, doi: [10.1016/j.autcon.2011.02.002](https://doi.org/10.1016/j.autcon.2011.02.002).
- [8] L. Czarnecki and A. Garbacz, *Advances in Material Sciences and Restoration No. 2 Adhesion in Interfaces of Building Materials: a Multi-scale Aproah*. Aedificatio Publishers, 2007.
- [9] T. Mathia, F. Louis, G. Maeder, and D. Mairey, "Relationship between surface states, finishing, processes and engineering properties," *Wear*, vol. 83, pp. 241–250, 1982.
- [10] N.B. Winter, *Scanning Electron Microscopy of Cement and Concrete*. Woodbridge, UK: WHD Microanalysis Consultants Ltd, Rendlesham, 2012.
- [11] K. Scrivener, R. Snellings, and B. Lothenbach, *A Practical Guide to Microstructural Analysis of Cementitious Materials*. CRC Press Taylor & Francis Group, 2016.
- [12] A. Saran A.S and M. Palanisamy, "Concrete Microstructure – A Review," *Imp. J. Interdiscip. Res.*, vol. 2, no. 12, pp. 1670–1673, 2016.
- [13] L. Czarnecki *et al.*, "Research of the Frost Resistance Durability of Concrete Material Road Engineering," *Bull. Pol. Acad. Sci. Tech. Sci.*, vol. 65, no. 6, pp. 1328–1331, 2016, doi: [10.14359/9782](https://doi.org/10.14359/9782).

- [14] I.N. Grubeša, B. Markovic, M. Vracevic, M. Tunkiewicz, I. Szenti, and Á. Kukovecz, "Pore structure as a response to the freeze/thaw resistance of mortars," *Materials (Basel)*, vol. 12, no. 19, pp. 1–16, 2019, doi: [10.3390/ma12193196](https://doi.org/10.3390/ma12193196).
- [15] Z. Jiang, B. He, X. Zhu, Q. Ren, and Y. Zhang, "State-of-the-art review on properties evolution and deterioration mechanism of concrete at cryogenic temperature," *Constr. Build. Mater.*, vol. 257, p. 119456, Oct. 2020, doi: [10.1016/j.conbuildmat.2020.119456](https://doi.org/10.1016/j.conbuildmat.2020.119456).
- [16] M. Pigeon, J. Marchand, and R. Pleau, "Frost resistant concrete," *Constr. Build. Mater.*, vol. 10, pp. 339–348, 1996, doi: [10.1016/0950-0618\(95\)00067-4](https://doi.org/10.1016/0950-0618(95)00067-4).
- [17] S.H. Dong, D.C. Feng, S.H. Jiang, and W.Z. Zhu, "Effect of Freezing Temperature on the Microstructure of Negative Temperature Concrete," *Adv. Mater. Res.*, vol. 663, pp. 343–348, Feb. 2013, doi: [10.4028/www.scientific.net/AMR.663.343](https://doi.org/10.4028/www.scientific.net/AMR.663.343).
- [18] J. Guo, W. Sun, Y. Xu, W. Lin, and W. Jing, "Damage Mechanism and Modeling of Concrete in Freeze–Thaw Cycles: A Review," *Buildings*, vol. 12, no. 9, p. 1317, 2022.
- [19] Y. Liu, B. Jin, J. Huo, and Z. Li, "Effect of microstructure evolution on mechanical behaviour of concrete after high temperatures," *Mag. Concr. Res.*, vol. 70, no. 15, pp. 770–784, 2018, doi: [10.1680/jmacr.17.00197](https://doi.org/10.1680/jmacr.17.00197).
- [20] K. Schabowicz, T. Gorzelańczyk, Ł. Zawislak, and F. Chyliński, "Influence of Fire Exposition of Fibre-Cement Boards on Their Microstructure," *Materials (Basel)*, vol. 16, no. 18, p. 6153, Sep. 2023, doi: [10.3390/ma16186153](https://doi.org/10.3390/ma16186153).
- [21] Y. Li, Z. Liu, and J. Jiang, "Microstructure and macroscopic properties of low-carbon concrete subjected to elevated temperature: State-of-the-Art Review," *J. Build. Eng.*, vol. 86, p. 108731, Jun. 2024, doi: [10.1016/j.jobbe.2024.108731](https://doi.org/10.1016/j.jobbe.2024.108731).
- [22] N. Gowripalan, J.G. Cabrera, A.R. Cusens, and P.J. Wainwright, "Effect of curing on durability," *Concr. Int.*, vol. 12, no. 2, pp. 47–54, 1990.
- [23] J.M. Vandenbossche, *A Review of the Curing Compounds and Application Techniques Used by the Minnesota Department of Transportation for Concrete Pavements*. Minnesota, USA: Minnesota Department of Transportation Office of Research Services, 1999.
- [24] K. Guo, G. Zhang, Y. Li, J. Yang, and Q. Ding, "The mechanism of curing regimes on the macroscopic properties and microstructure of ultra-high performance concrete with lightweight aggregates," *J. Build. Eng.*, vol. 82, p. 108236, Apr. 2024, doi: [10.1016/j.jobbe.2023.108236](https://doi.org/10.1016/j.jobbe.2023.108236).
- [25] F. Chyliński, A. Michalik, and M. Kozicki, "Effectiveness of Curing Compounds for Concrete," *Materials (Basel)*, vol. 15, no. 7, p. 2699, Apr. 2022, doi: [10.3390/ma15072699](https://doi.org/10.3390/ma15072699).
- [26] J. Wang *et al.*, "Influence of rapid curing methods on concrete microstructure and properties: A review," *Case Stud. Constr. Mater.*, vol. 17, no. October, p. e01600, 2022, doi: [10.1016/j.cscm.2022.e01600](https://doi.org/10.1016/j.cscm.2022.e01600).
- [27] W. Zhao, Z. Fan, X. Li, L. Kong, and L. Zhang, "Characterization and Comparison of Corrosion Layer Microstructure between Cement Mortar and Alkali-Activated Fly Ash/Slag Mortar Exposed to Sulfuric Acid and Acetic Acid," *Materials (Basel)*, vol. 15, no. 4, p. 1527, 2022, doi: [10.3390/ma15041527](https://doi.org/10.3390/ma15041527).
- [28] J. Witkowska-Dobrev *et al.*, "Effect of different water-cement ratios on the durability of prefabricated concrete tanks exposed to acetic acid aggression," *J. Build. Eng.*, vol. 78, p. 107712, Nov. 2023, doi: [10.1016/j.jobbe.2023.107712](https://doi.org/10.1016/j.jobbe.2023.107712).
- [29] L. Czarnecki and P. Woyciechowski, "Modelling of concrete carbonation; is it a process unlimited in time and restricted in space?," *Bull. Pol. Acad. Sci. Tech. Sci.*, vol. 63, no. 1, pp. 43–54, Mar. 2015, doi: [10.1515/bpasts-2015-0006](https://doi.org/10.1515/bpasts-2015-0006).
- [30] D. Józwiak-Niedźwiedzka, M. Sobczak, and K. Gibas, "Carbonation of concretes containing calcareous fly ashes," *Roads Bridg. – Drogi i Mosty*, vol. 12, no. 2, pp. 223–236, 2013, doi: [10.7409/rabdim.013.016](https://doi.org/10.7409/rabdim.013.016).
- [31] L. Luo and W. Yao, "Effect of chloride ion corrosion on the microstructure of multiple interfaces of alkali-activated GGBS/FA based recycled concrete," *J. Build. Eng.*, vol. 95, p. 110270, Oct. 2024, doi: [10.1016/j.jobbe.2024.110270](https://doi.org/10.1016/j.jobbe.2024.110270).
- [32] V.S. Ramachandran, R.M. Paroli, J.J. Beaudoin, and A.H. Delgado, "Handbook of Thermal Analysis of Construction Materials (Building Materials Series)," *Noyes Publ.*, p. 680, 2002, doi: [10.1016/B978-081551487-9.50002-5](https://doi.org/10.1016/B978-081551487-9.50002-5).
- [33] F.W. Wilburn, "Handbook of Thermal Analysis of Construction Materials," *Thermochim. Acta*, vol. 406, no. 1–2, p. 249, 2003, doi: [10.1016/S0040-6031\(03\)00230-2](https://doi.org/10.1016/S0040-6031(03)00230-2).
- [34] P. Falaciński, A. Machowska, and Ł. Szarek, "The impact of chloride and sulphate aggressiveness on the microstructure and phase composition of fly ash-slag mortar," *Materials (Basel)*, vol. 14, no. 16, p. 4430, 2021, doi: [10.3390/ma14164430](https://doi.org/10.3390/ma14164430).
- [35] F. Han, Z. Zhang, J. Liu, and P. Yan, "Effect of water-to-binder ratio on the hydration kinetics of composite binder containing slag or fly ash," *J. Therm. Anal. Calorim.*, vol. 128, no. 2, pp. 855–865, May 2017, doi: [10.1007/s10973-016-6015-4](https://doi.org/10.1007/s10973-016-6015-4).
- [36] A. Runci, J. Provis, and M. Serdar, "Microstructure as a key parameter for understanding chloride ingress in alkali-activated mortars," *Cem. Concr. Compos.*, vol. 134, no. October, p. 104818, 2022, doi: [10.1016/j.cemconcomp.2022.104818](https://doi.org/10.1016/j.cemconcomp.2022.104818).
- [37] J. Ying and X. Xi, "Microstructure and Chloride Diffusion Properties of Hardened Fly Ash Cement Paste with Three-dimensional Graphene," *Int. J. Concr. Struct. Mater.*, vol. 16, no. 1, p. 8, 2022, doi: [10.1186/s40069-021-00494-5](https://doi.org/10.1186/s40069-021-00494-5).
- [38] P.C. Adtın, "Cements of yesterday and today – concrete of tomorrow," *Cement Concr. Res.*, vol. 30, no. 9, pp. 1349–1359, 2000, doi: [10.1016/S0008-8846\(00\)00365-3](https://doi.org/10.1016/S0008-8846(00)00365-3).
- [39] L. Czarnecki, "Sustainable concrete; is nanotechnology the future of concrete polymer composites?," *Adv. Mater. Res.*, vol. 687, no. April, pp. 3–11, 2013, doi: [10.4028/www.scientific.net/AMR.687.3](https://doi.org/10.4028/www.scientific.net/AMR.687.3).
- [40] K.P. Bautista-Gutierrez, A.L. Herrera-May, J.M. Santamaría-López, A. Honorato-Moreno, and S.A. Zamora-Castro, "Recent progress in nanomaterials for modern concrete infrastructure: Advantages and challenges," *Materials (Basel)*, vol. 12, no. 21, pp. 1–40, 2019, doi: [10.3390/ma12213548](https://doi.org/10.3390/ma12213548).
- [41] C.S.R. Indukuri, R. Nerella, and S.R.C. Madduru, "Effect of graphene oxide on microstructure and strengthened properties of fly ash and silica fume based cement composites," *Constr. Build. Mater.*, vol. 229, p. 116863, 2019, doi: [10.1016/j.conbuildmat.2019.116863](https://doi.org/10.1016/j.conbuildmat.2019.116863).
- [42] Q.-H. Luo and S.-E. Fang, "Modified natural seawater sea-sand concrete: Linking microstructure to mechanical performance," *J. Build. Eng.*, vol. 98, p. 111461, Dec. 2024, doi: [10.1016/j.jobbe.2024.111461](https://doi.org/10.1016/j.jobbe.2024.111461).

- [43] O. Szlachetka, J. Witkowska-Dobrev, F. Chyliński, M. Dohojda, and B. Francke, "Effect of seawater on concrete elements," *4th International Conference Strategies toward Green Deal Implementation Water, Raw Materials & Energy*, 2023.
- [44] P.E. Grattan-Bellew, "Microstructural investigation of deteriorated Portland cement concretes," *Constr. Build. Mater.*, vol. 10, no. 1, pp. 3–16, Feb. 1996, doi: [10.1016/0950-0618\(95\)00066-6](https://doi.org/10.1016/0950-0618(95)00066-6).
- [45] R. Wang, "The Role of Polymer in Calcium Sulfoaluminate Cement-Based Materials," *Concrete-Polymer Composites in Circular Economy. ICPIC 2023. Springer Proceedings in Materials*, 2025, vol. 61, pp. 171–180, doi: [10.1007/978-3-031-72955-3_16](https://doi.org/10.1007/978-3-031-72955-3_16).
- [46] P. Brown, R.D. Hooton, and B. Clark, "Microstructural changes in concretes with sulfate exposure," *Cem. Concr. Compos.*, vol. 26, no. 8, pp. 993–999, 2004, doi: [10.1016/j.cemconcomp.2004.02.033](https://doi.org/10.1016/j.cemconcomp.2004.02.033).
- [47] H.P. Maarten A.T.M. Broekmans, *Applied Mineralogy of Cement & Concrete*. Chantilly, Virginia: The Mineralogical Society of America, USA, 2018.
- [48] D. Józwiak-Niedźwiedzka, M. Dąbrowski, K. Gibas, A. Antolik, and M.A. Glinicki, "Alkali-silica reaction and microstructure of concrete subjected to combined chemical and physical exposure conditions," *MATEC Web Conf.*, vol. 163, p. 05009, Jun. 2018, doi: [10.1051/mateconf/201816305009](https://doi.org/10.1051/mateconf/201816305009).
- [49] R. Rodrigues, S. Gaboreau, J. Gance, I. Ignatiadis, and S. Betelu, "Reinforced concrete structures: A review of corrosion mechanisms and advances in electrical methods for corrosion monitoring," *Constr. Build. Mater.*, vol. 269, p. 121240, Feb. 2021, doi: [10.1016/j.conbuildmat.2020.121240](https://doi.org/10.1016/j.conbuildmat.2020.121240).
- [50] T.U. Mohammed, H. Hamada, A. Hasnat, and M.A. Al Mamun, "Corrosion of steel bars in concrete with the variation of microstructure of steel-concrete interface," *J. Adv. Concr. Technol.*, vol. 13, no. 4, pp. 230–240, 2015, doi: [10.3151/jact.13.230](https://doi.org/10.3151/jact.13.230).
- [51] H. Torbati-Sarraf and A. Poursaei, "Corrosion Improvement of Carbon Steel in Concrete Environment through Modification of Steel Microstructure," *J. Mater. Civ. Eng.*, vol. 31, no. 5, 2019, doi: [10.1061/\(asce\)mt.1943-5533.0002677](https://doi.org/10.1061/(asce)mt.1943-5533.0002677).