Proc. 1st International Conference on Structural Engineering Research (iCSER 2017)
20-22 Nov 2017, Sydney, Australia
ISBN: 978-0-6480147-6-8

Addressing the Durability Issues of Construction Materials using Microstructural Analysis

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Abstract

Different materials and structures are designed for a certain service life. However, integrity and durability of materials particularly, newly-developed or waste materials used in construction to make them environmentally friendly, can affect the performance of structures and the design life. This integrity could be in the materials from the beginning like porous recycled aggregates that may cause pops out in concrete. It also could happen during the service life due to different reasons such as exposure to aggressive environments or fire. Loss of integrity of the anode metal used in cathodic protection of concrete structures is an example of the later mentioned issue. Detecting the mechanism of defect of construction materials used, not only assists to improve the development of better future construction materials, but also assists with the repair of the defects. Microstructural analysis of samples is an effective method to assess the integrity of materials. It is used to determine practical solutions for the repair and remediation. This paper reveals the importance of use of microstructural analysis to evaluate materials used in structures through explanation of examples of the application of scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD) analysis methods and X-ray mapping (XRM) utilised in diagnosis of the construction materials in service.

***Keywords*:** Microstructural analysis, construction materials integrity, new building materials

1. INTRODUCTION

Characterisation is an essential aspect of materials research that involves the determination of point-to-point variation in composition, structure and microstructure of materials. Understanding the distribution of elements and phases in structures is critical to optimising the performance of all materials (Wuhrer, Moran et al. 2006). Apart from its applications in areas of the science, art, biology, medicine, forensic etc, there are many applications and possibilities for the use of microstructural analysis in the area of engineering materials. In this paper the most common types of instruments used in the microstructural study of building materials, including scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS), X-ray diffraction (XRD) and X-ray mapping (XRM), are introduced in addition to some examples of their applications in understanding the behaviour of building materials.

2. TECHNIQUES AND EXAMPLES

2.1. Scanning electron microscope with energy dispersive spectroscopy (SEM-EDS)

Scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) is one of the most useful instruments utilised to study the microstructural analysis of construction materials.

Two signals are measured in the SEM providing different information about the sample, namely, secondary electrons (SE) and back-scattered electrons (BSE). Secondary electron (SE) images provide information about the topography of samples, whereas back-scattered electron (BSE) images contain compositional information that is differentiated by their atomic numbers.

Choice of the detector type for SEM imaging depends on the purpose of the analysis. For example, if the chemical impurity and material defect is of interest in a problem, BSE could be more informative, whereas in case of studying the morphological integrity and investigation of microcracks, SE is of more value. On some occasions, the combination of the two is useful.

Energy dispersive spectrometry (EDS), more commonly known as microanalysis, is also an established technique for the analysis of the chemical composition of materials in a SEM. The EDS detector is mounted in the side of the SEM (Figure 1b), and collects characteristic X-rays emitted during interactions with the SEM electron beam and the sample. This signal is given as a spectrum from which the chemical composition of the sample can be determined and quantified.

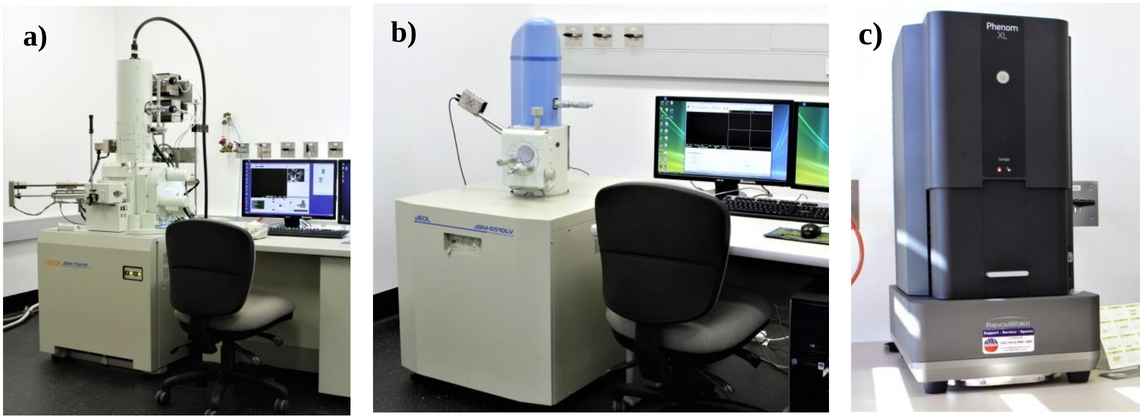

Figure 1. SEM and EDS, a) a JEOL 7001F (high vacuum SEM instrument equipped with Bruker Microanalysis EDS), b) JEOL JSM 6510 equipped with Moran-Amptek Microanalysis-EDS and c) a Phenom XL benchtop SEM/EDS

2.2. Detecting the defects and durability issues in building materials in service

Materials used in construction may have defects or impurity from the beginning or they can be affected by the environments they are exposed to. It may cause an undesirable appearance, not meeting the serviceability requirements and could even cause structural failure. Figure 2 shows some examples of the defects and durability issues in construction materials.

Figures 3(a-c) show a concrete deck that has gone through durability issues. The concrete cover has been degraded and there was no cover left for the reinforcing bars in some areas. Even the reinforcing

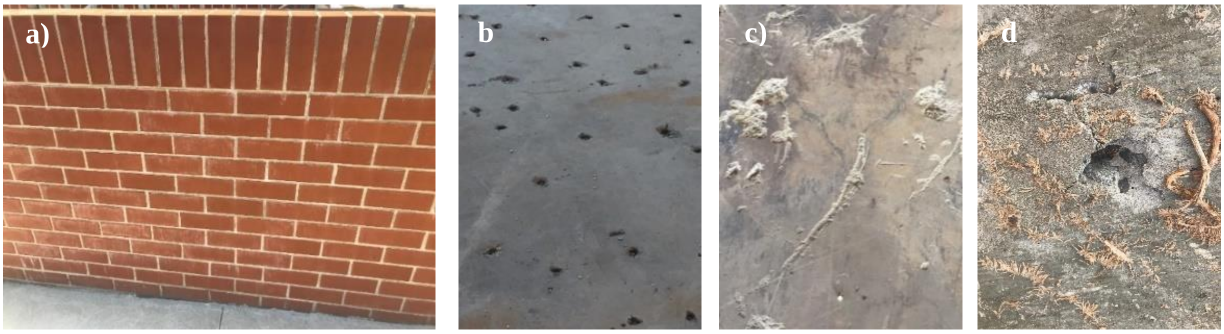

Figure 2. a) Efflorescence defect on brick walls, (b,c) defects in concrete floor and d) reinforced concrete degradation underneath a slab

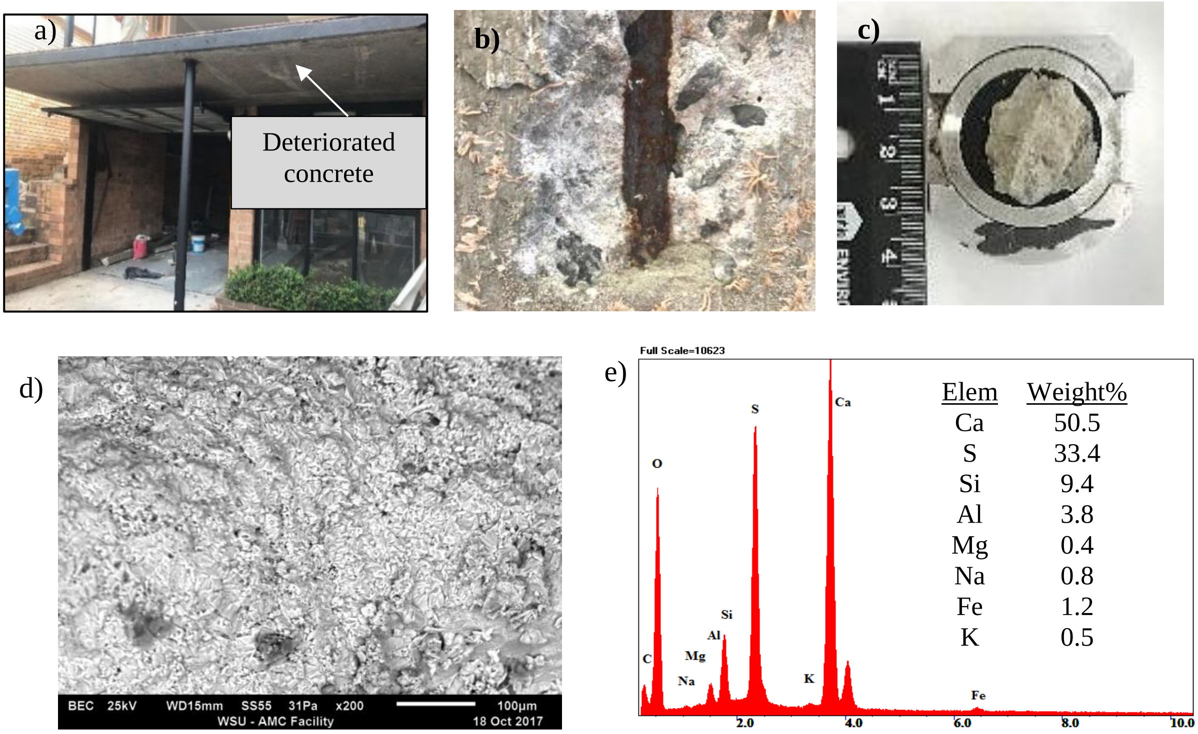

Figure 3. a) Defect under the balcony's reinforced concrete slab, b) corrosion of reinforcing bar and c) mounted sample for SEM/EDS analysis, d) SEM and e) EDS analysis of the deteriorated area of the concrete balcony slab

Bars covered by concrete, had no strength in the structure. To find the reason for this issue, samples were taken from the concrete adjacent to the reinforcing bars. They were examined by SEM and EDS. Figure 3c shows a sample mounted on a SEM stub to investigate the issue.

The results of SEM and EDS analysis are shown in Figure 3d and 3e. As shown, high amounts of sulphur (33.4%, whereas we do not expect more than 2% sulphur in Portland cement) has caused the degradation of concrete cover. Exposure of reinforcement to the air and lack of the protective alkaline layer (concrete) for the reinforcement, led to corrosion of reinforcing bars. This sulphur can come from different sources that is out of the scope of this paper.

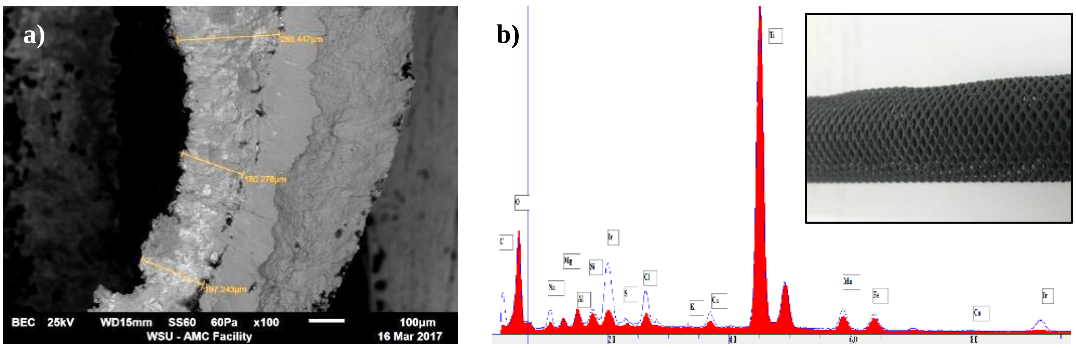

Figure 4. Investigation of a used anode in a cathodic protection system a) SEM image of its thickness and b) EDS analysis of intact and degraded section

Figure 4 shows SEM and EDS analysis of an anode used in cathodic protection of reinforced concrete structures. As shown the integrity of the anode was examined through measuring the thickness of cross section of the sample as well as EDS analysis of different areas (Figure 4b). EDS analysis showed different amounts of Iridium at degraded areas compared to intact areas. Although by optical observation the anode looked sound, from the analysis it was concluded that they needed to be replaced.

2.3. X-ray diffraction (XRD) and its application in building materials study

X-ray diffraction (XRD) is a powerful technique for characterizing crystalline materials that can provide information on crystallinity, phases, preferred crystal orientation, and other structural parameters. X-ray diffraction peaks are produced by constructive interference of a monochromatic beam of x-rays scattered at specific angles from each set of lattice planes in a sample and finally, the phases are identified by searching the standard data base. Figure 5 shows a Bruker XRD instrument and different types of sample preparation for XRD analysis.

Figure 5. a) X-ray diffraction (XRD) instrument and b) sample preparation for the XRD

Depending on the material type, different methods of sample preparation can be used for the XRD analysis. This analysis is very helpful in detecting the new phases resulting from the chemical reactions of corrosive agents to building materials such as concrete.

For example, it is known that Portlandite (Ca $(OH)_2$) in the cement reacts with the sulphur environments such as sewage systems, and forms calcium sulphate (gypsum). XRD reveals phases present in a material, as shown in Figure 6, allowing for the mechanism and sources of deterioration to be identified. In Figure 6, a conventional concrete has been tested before and after exposure to 13% sulphuric acid solution for 4 weeks. Gypsum peaks were observed in the XRD spectra after acid exposure.

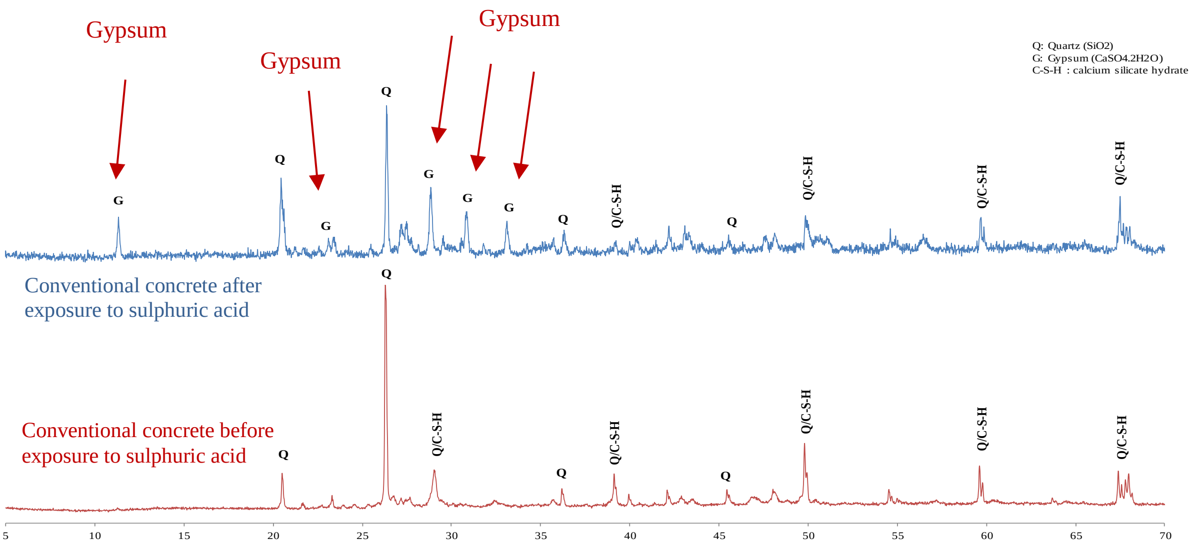

Figure 6. Formation of gypsum in conventional concrete after 4 weeks of exposure to 13% sulphuric acid (Salek, 2016)

Microstructural techniques are also used in the development of new construction material, and can help to predict their performance in real applications (e.g. when subjected to fire). Use of recycled aggregate in concrete, geopolymer or alkali activated concrete and use of wastes like glass and rubber in concrete are few examples of these new materials to move toward more sustainable building materials.

Figure 7 shows the development of phases in a semi geopolymer concrete over time. The sample was mixed and initially analysed by XRD. A new XRD analysis was then performed every hour. Some examples of these analysis are shown in Figure 7 at selected times as an example. As can be seen, phases change over time, some of which are desirable in geopolymer concrete such as muscovite, hematite, kaolinite and zeolite ((Ke, Bernal et al. 2015, Hajimohammadi and van Deventer 2017).

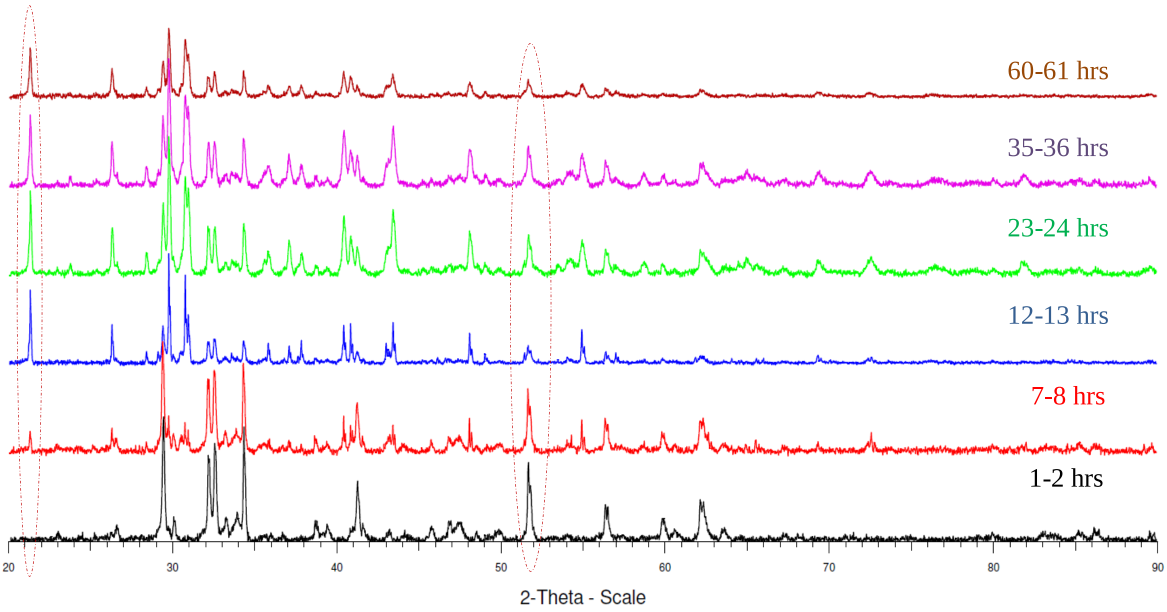

Figure 7. XRD analysis in identification of phases in semi geopolymer concrete starting from the hydration till 3 days every hour.

2.4. X-ray mapping and building materials investigation

X-ray mapping (XRM) is used for identifying the location of individual elements and mapping the spatial distribution of specific elements and phases within a sample (Wuhrer, Moran et al. 2006). Pseudo colouring is a method for determining elemental associations. In this technique, three elemental maps are assigned the colours red, green, and blue (Moran and Wuhrer (2010), (Wuhrer and Moran 2015)). XRM and detecting the distribution of elements in a sample reveals important facts regarding different samples.

Figure 8a shows the super probe used for this purpose and Figure 8b shows a sample prepared for the XRM. Figure 8c shows an example of pseudo colour image of interface of a concrete sample after exposure to sulphuric acid as an example. In this image where is red is rich with sulphur, where is blue has higher silica and the area which is yellow (between red and green based on the scale bar below the image) is a phase containing both calcium and sulphur (Salek 2016).

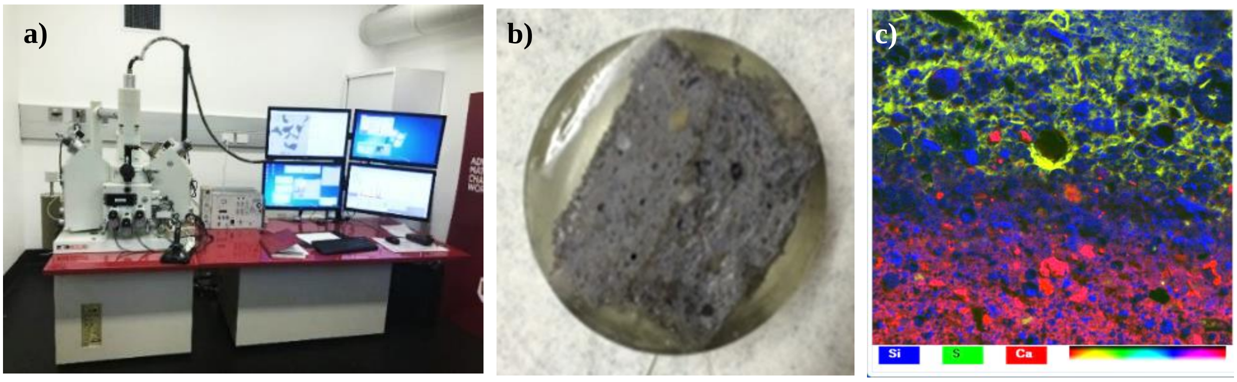

Figure 8. a) A JEOL JXA-8600 super probe used for X-ray mapping, b) a prepared sample for XRM analysis and c) Pseudo colour image from a concrete sample exposed to sulphuric acid.

3. CONCLUSION

Microstructural analysis has an extensive application in the identification of reasons for durability issues or defects in building construction materials. It is also beneficial in the development of new construction materials by investigation of their microstructure and prediction of their behaviour when subjected to different conditions, such as fire or exposure to harsh environments.

SEM equipped with the EDS and XRD analysis are the most commonly used methods for microanalsyis. The combined use of these techniques provides a powerful, complimentary assessment of construction materials.

4. ACKNOWLEDGMENTS

The authors would like to acknowledge the support of Advanced Materials Characterisation Facility (AMCF) and the Centre for Infrastructure Engineering at Western Sydney University (CIE). We would also like to thank Ms. Mahya Askarian and Mr. Michael van Koeverden for use of their data and photos in this paper.

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