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学 位 論 文 内 容 の 要 旨

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学 位 論 文 題 名

Application of X-ray CT to the study of microstructure and diffusivity in cementitious materials
(X 線 CT を用いたセメント硬化体の微細構造と拡散性状に関する研究)

At the beginning of its development, the X-ray CT technique originally was developed for medical analysis. However, along with the advances in technology, the ability of X-ray CT continues to increase. Therefore, the use of X-ray CT is no longer intended for medical application but has expanded to other fields such as civil engineering especially for material science. Related to construction materials, there are many experimental methods that can be used to study the microstructure of cementitious materials such as scanning electron microscope (SEM), backscattered electron (BSE), mercury intrusion porosimetry (MIP) and permeability test. However, each of those experimental methods generally only provides few aspects related to the microstructure of cementitious materials. On the other hand, there are many aspects which can be obtained from the microstructure of cementitious materials just through the use of X-ray CT technique. This present study describes the versatility of the application of X-ray CT technique to study the microstructure and diffusivity of cementitious materials. In this research, there are two types of X-ray CT systems used to acquire the 3D image of the internal structure of cementitious materials i.e. synchrotron X-ray CT with spatial resolution of $0.5\ \mu\text{m}$ and micro-focus X-ray CT with lower resolution ($>10\ \mu\text{m}/\text{pixel}$).

The first and arguably the most important in the use of X-ray CT is the observation of the three-dimensional pore structure of the hardened cementitious materials. The application of X-ray CT certainly can be expanded through the use of different types of cementitious materials as well as its curing period. In this case, cement paste specimen mixed with silica fume and fly ash (low alkali binder) was used and (Ordinary Portland Cement) OPC cement paste was prepared as a comparison. The pore structure parameters such as ash total porosity, connectivity, and pore size distribution were obtained through the analysis of 3D microtomographic images. These 3D pore structure parameters for all specimens decreased with increasing curing periods but with different rate. In addition, the application of synchrotron X-ray CT allows us to investigate solid phases in the microstructure of cementitious materials through the analysis of its linear attenuation coefficient (LAC). The difference in the change of solid phases in both types of cementitious materials due to increasing curing periods was observed through the change of LAC histogram.

On the other hand, despite having a lower resolution, the use of micro-focus X-ray CT was intended to identify the larger void space in cementitious materials such as cracks and air voids and also allows us to use the specimens with larger size. In this way, the scope of the observation using X-ray CT was expanded. Micro-focus X-ray CT was employed to observe cracked mortar specimens due to mechanical loading. The cracks in beam specimens of the size of $10 \times 20 \times 60\ \text{mm}$ and cylinder specimens of the size of $20\ \text{mm}$ in diameter and $40\ \text{mm}$ in height were induced by flexural test and splitting tensile test, respectively. Subsequently, the 3D crack geometry was obtained from microtomographic images. Through the analysis of 3D crack geometry, the crack geometry parameters such as tortuosity and constrictivity were obtained. The tortuosity of flexural crack and splitting tensile crack were found to be around 1.15 and 1.25, respectively, and it was independent to the crack opening width and whether fly ash was added or not. Meanwhile, it is found that the type of cracks and materials play an important role on the constrictivity of the crack. The constrictivity increased for fly ash mortar having roughly the equivalent crack opening width of OPC mortar.

The scope of the observation using X-ray CT was expanded even further. The application of micro-focus X-ray CT coupled with in-situ tracer diffusion test was conducted to clarify the transport mech-

anism in cracked mortar specimens. It is found that the diffusivity in the crack was controlled by following factors: the crack opening width, its tortuosity and constrictivity and the degree of water saturation within the crack space. In this regard, a diffusion-accessible path factor was proposed to account for the degree of water saturation within the crack space. The diffusion coefficient along the crack of cracked specimen in oven-dried condition was much more greater than that of partially saturated condition under the same crack opening width. In addition, it was confirmed that the addition of fly ash could reduce the diffusivity through the uncracked body as compared with normal OPC mortar of equivalent diffusion coefficient along the crack.

Furthermore, the variations in cementitious materials were added. In this regard, deteriorated specimens were used in X-ray CT observation. There were three types of deterioration applied in the specimen namely leaching, mechanical loading and high temperature exposure followed by re-curing. X-ray CT technique was used to observe the change in microstructure in cementitious materials as the effect of deterioration. As for leaching, after the observation using synchrotron X-ray CT and image analysis, the total porosity and percolated porosity of normal cement paste specimen significantly increased after leaching test as compared to that of cement paste specimen with low alkali binder. Accordingly, the distribution of pore structure of normal cement paste specimen was significantly changed as compared to that of cement paste specimen with low alkali binder. Meanwhile, the change on solid phases at each specimen due to leaching test was successfully represented through the change of the LAC histograms.

In the study of deteriorated cementitious materials due to mechanical loading, synchrotron X-ray CT was combined with micro-tensile instrument and the observation during the application of load was conducted in-situ inside synchrotron X-ray CT chamber. Although there was no definite result of this experiment, however, by improving the quality both sample preparation and loading instrument, the behavior of microstructure under external load can be observed through the application of synchrotron X-ray CT combined with other mechanical tests.

Using micro-focus X-ray CT, concrete specimen in three different conditions, namely before heating, after heating and re-curing was examined. It was observed that cracks occurred in the concrete specimen due to heating can be categorized into three types of cracks, namely interfacial cracks between mortar and coarse aggregates, bridging cracks between aggregates and bridging cracks between aggregates and air voids. Therefore, the volume of total and percolated porosity of heated specimen increased due to the presence of these cracks. However, the volume of total and percolated porosity decreased as the result of water re-curing for 28 days. In addition, the pore size distribution became finer after 28 days re-curing period as compared with that of after heating condition.

Through the application of X-ray CT technique this research could provide better understanding of the microstructure change in cementitious materials. In addition, with the proposed X-ray CT technique coupled with in-situ tracer diffusion test, the transport mechanism of diffusion inside crack and through the uncracked matrix would be further understood. Therefore, this research can contribute to the clarification of durability aspect and to more accurate prediction of service life for concrete structures.