

マテリアル先端リサーチインフラ利用報告書

ARIM User's Report

[Release : 2024.07.25] [Update : 2025.04.07]

課題データ / Project Data

課題番号 Project Issue Number	23QS0020
利用課題名 Title	Phase evolution, mechanical properties, and morphology of cementitious silicate hydrates subjected to carbonation
利用した実施機関 Support Institute	量子科学技術研究開発機構 / QST
機関外・機関内の利用 External or Internal Use	外部利用/External Use
ARIM半導体基盤PF 関連課題 Related to ARIM-SETI	指定なし / No Designation
横断技術領域 Cross-Technology Area	計測・分析/Advanced Characterization 物質・材料合成プロセス/Molecule & Material Synthesis
重要技術領域 Important Technology Area	マテリアルの高度循環のための技術/Advanced materials recycling technologies
キーワード Keywords	セメント材料,局所構造,X線回折/ X-ray diffraction,放射光/ Synchrotron radiation

利用者と利用形態 / User and Support Type

利用者名 (課題申請者) User Name (Project Applicant)	Bae Sungchul
所属名 Affiliation	Department of Architectural Engineering, Hanyang University
共同利用者氏名 Names of Collaborators Excluding Supporters in the Hub and Spoke Institutes	兼松 学, Sumin Im, Gyeongryul Kim, Seongmin Cho, Jin Yun, Junho Kim, 細川 隆行, 堀合 航太
ARIM実施機関支援担当者 Names of Supporters in the Hub and Spoke Institutes	町田 晃彦
利用形態 Support Type	共同研究/Joint Research

利用した主な設備 / Equipment Used in This Project

利用した主な設備 Equipment ID & Name	QS-222 : 高速2体分布関数計測装置
---------------------------------	-----------------------

報告書データ / Report

<p>概要 (目的・用途・実施内容) Abstract (Aim, Use Applications and Contents)</p>	<p>To reduce CO₂ emissions, it is essential to applicate supplementary cementitious materials (SCMs), such as fly ash, granulated blast-furnace slag, and silica fume, that can partially replace cement. Despite the diversification in cement composition, the volume fraction of calcium-silicate-hydrate (C-S-H) still accounts for more than approximately 50% of the total composition of cement hydration products. <u>Due to the abundance of elements capable of modifying the C-S-H structure in SCMs, such as aluminum and magnesium, there are various types of silicate hydrates in cementitious materials (Fig.1): aluminum-incorporated C-S-H (C-A-S-H), magnesium-incorporated C-S-H (M-C-S-H), and magnesium silicate hydrate (M-S-H), respectively. These silicate hydrates affect the mechanical properties of cement paste according to the ratios of constituent elements (i.e., Ca/Si, Al/Si, Mg/Si, and Ca/Mg) [1].</u> There are three distinct periods during C-S-H carbonation, including a dissolution period, a diffusion period, and a slowly ongoing reaction period. During C-S-H carbonation under atmospheric conditions, C-S-H gel transforms into an amorphous silica gel and various polymorphs of calcium carbonate, such as amorphous calcium carbonates, calcite, vaterite, and aragonite [3]. <u>Due to the heterogeneous and amorphous characteristics of C-S-H gel, the carbonation kinetics of interatomic structure of C-S-H and the correlation between chemical decomposition and mechanical properties remain unknown [4].</u> Furthermore, the carbonation kinetics of other amorphous silicate hydrates in PC-based materials with SCMs, such as C-A-S-H and M-S-H, still remain unclear. Therefore, this study aims to investigate the carbonation kinetics of silicate hydrates and the relationship between interatomic structural changes and mechanical properties of amorphous silicate hydrates after the early stage of carbonation via <i>in-situ</i> loading synchrotron X-ray scattering experiments.</p>
<p>実験 Experimental</p>	<p>準備した合成 C-S-H, M-C-S-H, M-S-H, and C-A-S-H pasteに圧縮負荷をかけながら (常温) XRD実験を行う。事前に準備した試験片は 持ち込み装置である小型引張圧縮試験機一式 (AC 100 V) を用いて、予備実験の結果より最大15~18 MPaまで0.5~1 MPa間隔で圧縮応力を負荷した圧縮試験を行う予定とした。BL22XUのSi (111)二結晶分光器で単色化した70 keVのX線を使用する。検出器には回折計に搭載された大型デジタル2次元X線検出器を用い、入射ビームサイズは0.5 mm × 0.5 mmとした。試験片からの距離Lを304 mmおよび654 mmでそれぞれPDFおよびXRD解析用のデータを収集した。</p>
<p>結果と考察 Results and Discussion</p>	<p>After 10 min of carbonation, the peaks of MgCO₃ and CaCO₃ were detected as shown in the total X-ray scattering patterns. Furthermore, the intensity of the hump within the Q ranging from 1.5 to 2 Å increased, indicating that the depolymerization behavior of silicate chains in M-C-S-H was more apparent than that of C-S-H pastes. The interatomic distance at 2.4 Å regarding the calcium layers slightly decreased while that of the M-C-S-H phases almost unchanged after 30 min of carbonation. The peak intensity of interatomic distance between Mg and O increased. The PDF peaks according to the interatomic distance of Ca-Si (3.6 Å) decreased while that of M-C-S-H phase increased. These results show that the structural transformation from chain-like structure to plate-like structure due to the Mg substitution can reduce the decalcification behavior of C-S-H under carbon storage conditions.</p>

<p>図・表・数式 1 Figures, Tables and Equations 1</p>	<p>Pair distribution functions of (A) C-S-H pastes and (B) M-C-S-H pastes after carbonation curing time of 10, 20, and 30 min</p>
<p>その他・特記事項 (参考文献・謝辞等) Remarks(References and Acknowledgements)</p>	<p>Reference [1] E. Bernard, H. Nguyen, Magnesium silicate hydrate (M-S-H) stability under carbonation, <i>Cem Concr Res</i> 178 (2024) 107459. https://doi.org/10.1016/J.CEMCONRES.2024.107459. [2] D. Bonen, M.D. Cohen, Magnesium sulfate attack on Portland cement paste – II. Chemical and mineralogical analyses, <i>Cem Concr Res</i> 22 (1992) 707–718. https://doi.org/10.1016/0008-8846(92)90023-O. [3] J.L. García Calvo, A. Hidalgo, C. Alonso, L. Fernández Luco, Development of low-pH cementitious materials for HLRW repositories: Resistance against ground waters aggression, <i>Cem Concr Res</i> 40 (2010) 1290–1297. https://doi.org/10.1016/J.CEMCONRES.2009.11.008.</p> <p>Acknowledgments This study was supported by the National Research Foundation of Korea grants funded by the Korean government (MSIT) (Grant numbers NRF-2022R1A2C2010350).</p>

成果発表・成果利用 / Publication and Patents

<p>DOI (論文・プロシーディング) [1] DOI (Publication and Proceedings)</p>	<p>Gyeongryul Kim, Evaluation of the thermal stability of metakaolin-based geopolymers according to Si/Al ratio and sodium activator, <i>Cement and Concrete Composites</i>, 150, 105562(2024). DOI: 10.1016/j.cemconcomp.2024.105562</p>
--	--

口頭発表、ポスター発表 および、その他の論文[1] Oral Presentations etc.	Kim Gyeong Ryul, Cho Seong Min, Her Sung Wun, Yang Ji Hwan, Bae Sung Chul, "Assessment of High Temperature Resistance of Metakaolin-Based Geopolymer according to Si/Al Molar Ratios", Korea Concrete Institute 2023 Autumn Academic Conference (Jeju, Korea), 令和5年11月2日
口頭発表、ポスター発表 および、その他の論文[2] Oral Presentations etc.	Yun Jin, Bae Sungchul, "Nanoscale Structural Change of Calcium Silicate Hydrate Pastes upon Alkali Hydroxide Concentration", 2023 Korea Concrete Institute Autumn Conference (Seogwipo/Jeju/Republic of Korea), 令和5年11月2日, Grand Prize in the Graduation Thesis Category, 11th Outstanding Capstone Design and Graduation Thesis Competition, Korea Concrete Institute, 令和5年11月3日.
口頭発表、ポスター発表 および、その他の論文[3] Oral Presentations etc.	Yun Jin, Bae Sungchul, "Impact of Chloride and Sulfate Exposure on the Physicochemical Properties of Calcium Silicate Hydrate (C-S-H) at Various pH Levels", 2024 Spring Meeting of Korean Ceramic Society (Busan/Republic of Korea), 令和6年4月17日.
口頭発表、ポスター発表 および、その他の論文[4] Oral Presentations etc.	Im Sumin, Bae Sungchul, " An Analysis of Mechanical Behavior of Calcium Silicate Hydrates Incorporating Graphene Oxide", Architectural Institute of Korea 2024 Autumn Conference (Gyeongju/Republic of Korea), 令和6年10月24日.
口頭発表、ポスター発表 および、その他の論文[5] Oral Presentations etc.	Cho Seongmin, Bae Sungchul, "Analysis on the Atomistic- to Nano-scale Structure and Stress-strain Behavior of Carbonation Products of Calcium Silicate Minerals using High-energy X-ray", 2024 Fall Meeting of Korean Ceramic Society (Seoul/Republic of Korea), 令和6年10月18日..
特許出願件数 Number of Patent Applications	0件
特許登録件数 Number of Registered Patents	0件