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：2023.08.01】　【Update：2025.04.07】

課題データ / Project Data

課題番号 Project Issue Number	22QS0120
利用課題名 Title	載荷による加速炭酸化養生した純粋多様なカルシウムシリケート硬化体の反応生成物構造解析及び変形挙動特性解明
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
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 その他/Others
キーワード Keywords	Environmentally friendly material design, Nano-structural analysis,X線回折/X-ray diffraction,放射光/Synchrotron radiation

利用者と利用形態 / User and Support Type

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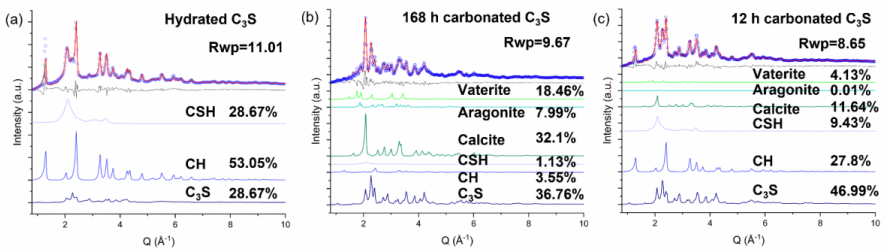
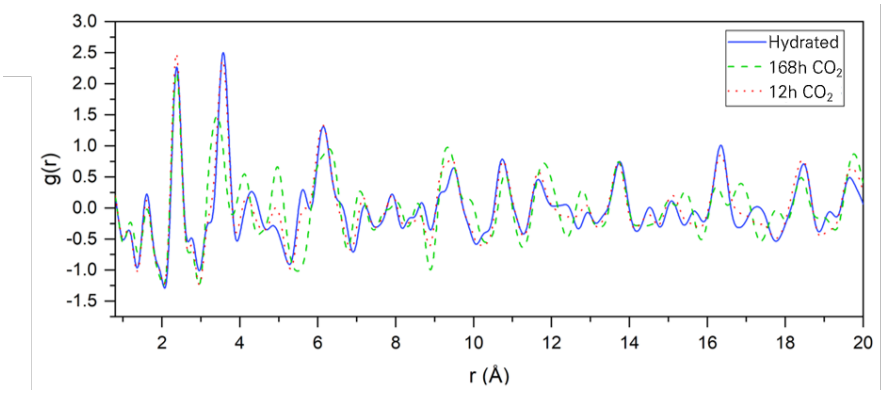
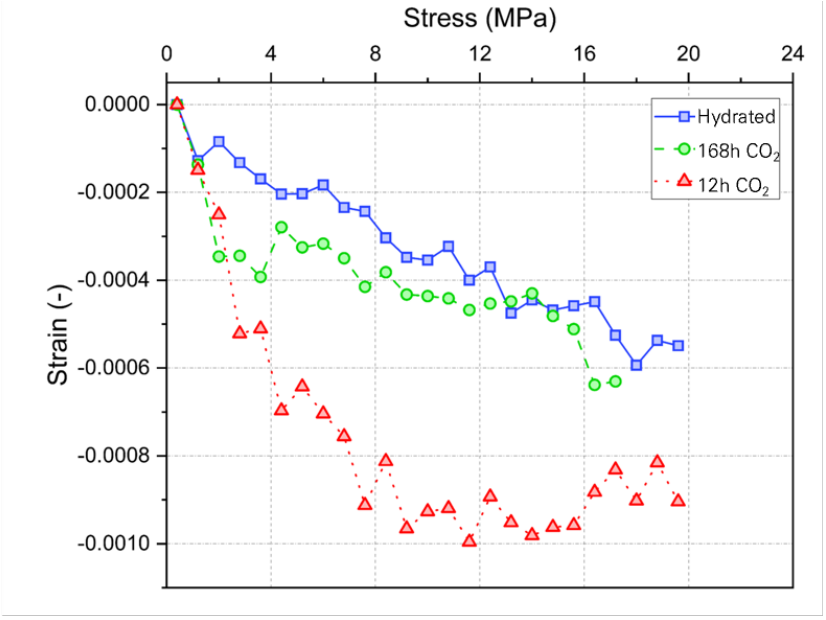
ARIM実施機関支援担当者 Names of Supporters in the Hub and Spoke Institutes	町田晃彦
利用形態 Support Type	共同研究/Joint Research

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

概要（目的・用途・実施内容） Abstract (Aim, Use Applications and Contents)	To compare the phase composition, load transferring mechanism, deformation behavior, and structural changes of pure calcium silicate minerals according to the Ca/Si ratio and carbonation period by X-ray diffraction and pair distribution function (PDF) analysis, high energy X-ray scattering experiment was performed on the 5x5x10 mm^3 sized paste mixture of C_3S, C_2S, C_3S_2, and CS (which corresponds to pure calcium silicate phases with varying chemical compositions (Ca/Si= 3, 2, 1.5, and 1) with the carbonation period of 0, 12, 24, 48, 72, 168 hours.
実験 Experimental	【利用した装置】：高速2体分布関数計測装置 【実験方法】 Based upon the experiments done earlier by the applicants, the experiments of carbonated calcium silicate pastes were performed in the form of paste mixtures (5×5×10 mm^3). First experiments were held by applying external loads during X-ray scattering experiments with the pure calcium silicate phases with varying chemical compositions (Ca/Si= 3, 2, 1.5, and 1, which corresponds to C_3S, C_2S, C_3S_2, and CS respectively). The samples were prepared by two types, hydrated and carbonated ones for the comparison of the results. The incident X-ray beam was targeted to the center of the sample in the size of 0.5 mm ×0.5 mm and the flat panel (FP) detector will be positioned at the distance of 304 and 654 mm from the pastes to achieve an extensive PDF Q-range and high angular resolution. External loads of 0.5 to 1MPa were applied each step until the rupture of the sample. The 3-dimensional internal microstructure of the samples weare further investigated by hard X-ray computed tomography at Pohang light source (PAL-7C beamline, X-ray energy: 8.3 keV (Ni K-edge)).
結果と考察 Results and Discussion	X-ray scattering results of the C_3S according to the initial carbonation period are displayed in Fig.1. The scattering peaks of the C_3S samples varies significantly by the initial carbonation period. Hydrated C_3S contains CSH and CH as a reaction product, whereas the carbonated C_3S shows decreased or no amount of CSH according to the Especially, the sample initially carbonated for 12 h (Fig. 1. (c)) shows distinct peaks indicating the presence of $Ca(OH)_2$ as a reaction product. With the increasing carbonation period, diffraction peak intensities of $CaCO_3$ significantly increases (Fig.1. (b)). PDF analysis results of the C_3S according to the initial carbonation period are displayed in Fig.2. The PDF peaks of the C_3S samples varies significantly by the initial carbonation period. The peaks located at 1.6, 2.4, 3.6 A is shifted according to the presence of carbonation products. Especially, the sample subjected to 168 h of initial carbonation exhibited the drastic changes compared to the one without carbonation curing. Atomistic strain calculated by the shifts of PDF peaks according to the constant step loading was shown in Fig. 3. The sample initially carbonated for 12 h had shown drastic strain at initial loading region, whereas the hydrated and 168 h carbonated samples had shown higher elastic modulus. Therefore, it was found that the longer the carbonation period becomes, the higher the elastic modulus became.
図・表・数式 1 Figures, Tables and Equations 1	 Fig. 1. X-ray scattering results of C_3S by varying initial carbonation period.

図・表・数式 2 Figures, Tables and Equations 2	 Fig. 2. PDF results of C_3S by varying initial carbonation period.
図・表・数式 3 Figures, Tables and Equations 3	 Fig. 3. Atomistic deformation behavior of C_3S by varying initial carbonation period.
その他・特記事項（参考文献・謝辞等） Remarks(References and Acknowledgements)	Sumin Im, Hyeonseok Jee, Heongwon Suh, Manabu Kanematsu, Satoshi Morooka, Hongbok Choe, Nishio Yuhei, Akihiko Machida, Jihoon Kim, Seungmin Lim, Sungchul Bae, "Insight on the mechanical properties of hierarchical porous calcium-silicate-hydrate pastes according to the Ca/Si molar ratio using in-situ synchrotron X-ray scattering and nanoindentation test", Construction and Building Materials, 365, 130034 (2023), DOI:10.1016/j.conbuildmat.2022.130034 Im Sumin, Bae Sungchul, "Multiscale investigation on the thermal stability of synthetic C-S-H pastes according to Ca/Si ratios", 16th International Congress on the Chemisstry of Cement, ICCC2023 (Bangkok, Thailand）, 令和5年9月19日 Kim Gyeongryul, Suh Heongwon, Cho Seungmin, Bae Sungchul, "Effect of the Formation of $MgCl_2$ based Magnesium Silicate Hydrate on the Physicochemical Characteristics of Calcium Silicate Hydrate", Korea Concrete Institute 2022 Spring Academic Conference (Seoul, Korea), 令和4年5月12日

成果発表・成果利用 / Publication and Patents

DOI（論文・プロシーディング）[1] DOI (Publication and Proceedings)	Seongmin Cho, Characteristic microstructural phase evolution and the compressive strength development mechanisms of tricalcium silicate pastes under various initial carbonation curing environments, *Construction and Building Materials*, **409**, 133866(2023). DOI: 10.1016/j.conbuildmat.2023.133866
口頭発表、ポスター発表および、その他の論文[1] Oral Presentations etc.	Im Sumin, Cho Seongmin, Chen Yukun, Bae Sungchul, "Microstructural Analysis of Calcium Silicate Hydrate (C-S-H) Incorporating Graphene Oxide via High Energy X-ray Scattering Measurement", Korea Concrete Institute 2023 Spring Academic Conference (Busan, Korea), 令和5年5月11日
口頭発表、ポスター発表および、その他の論文[2] Oral Presentations etc.	Cho Seongmin, Suh Heongwon, Kim Gyeongryul, Bae Sungchul, "Comparison of the nanostructure of carbonation products of hydraulic and non-hydraulic calcium silicates using pair distribution function", Architectural Institute of Korea 2023 Spring Conference (Seoul, Korea), 令和5年4月27日
口頭発表、ポスター発表および、その他の論文[3] Oral Presentations etc.	Cho, Seongmin, Im Sumin, Bae Sungchul, "Nano-structural Investigation of the Reaction Products of Pure Tricalcium Silicate Paste by Carbonation-hydration Curing Condition using Pair Distribution Function", Korea Concrete Institute 2022 Autumn Academic Conference (Jeju, Korea), 令和4年11月3日
口頭発表、ポスター発表および、その他の論文[4] Oral Presentations etc.	Cho Seongmin, Bae Sungchul,"An Experimental Study on the Micro-scale Deformation Behavior of CO2-Cured Di-calcium and Tri-calcium Silicate Pastes Using In-situ Loading Atomic Pair Distribution Function Analysis", The 1st RILEM International Conference on Mineral Carbonation for Cement and Concrete (Aachen/Germany), 令和6年4月17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