Experiments of CO_2 adsorption, CO_2 reactivity and structural stability of carbonate phases at high pressure and temperature conditions using synchrotron X-ray diffraction

David Santamaría-Pérez

VNIVERSITAT DE VALÈNCIA

ICMUV
INSTITUT DE CIÈNCIA DELS MATERIALS de la Universitat de València

MINISTERIO DE ECONOMÍA, INDUSTRIA Y COMPETITIVIDAD

GENERALITAT VALENCIANA
CONSELLERIA DE EDUCACIÓN, INVESTIGACIÓN, CULTURA Y DEPORTE

Source: National Institute of Standards and Technology

- CO_2 separation and storage in open frameworks

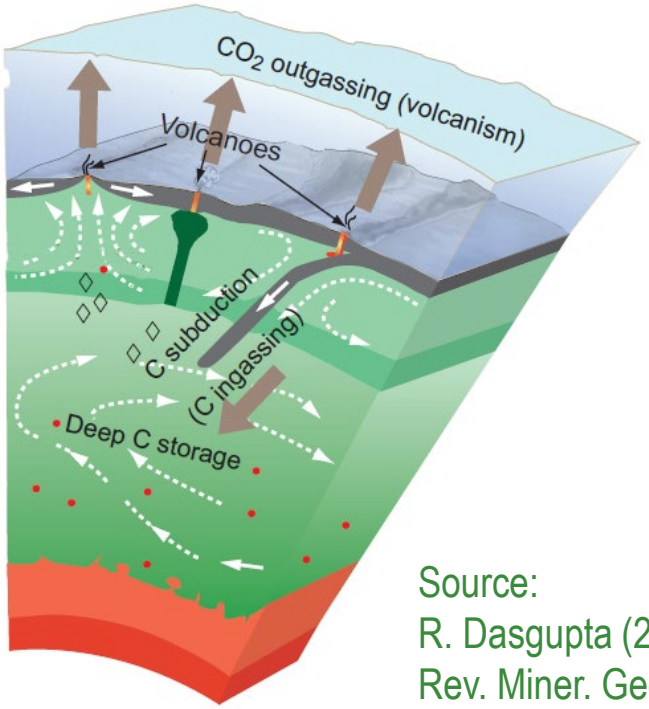
CO_2 trapping in zeolites

Data that could help designing potential CCS strategies

Further insight of the Earth's deep carbon cycle

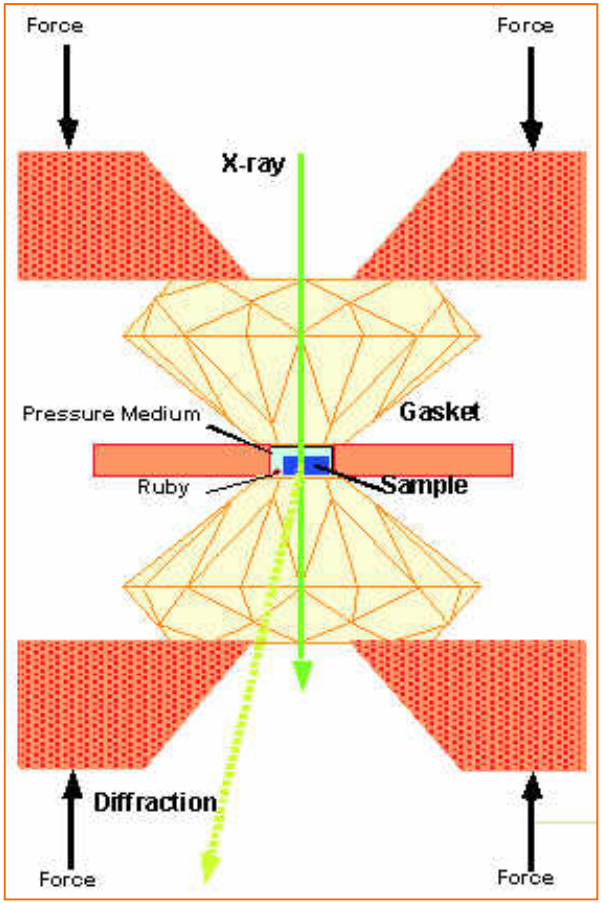
CO_2 sequestration as carbonate minerals

- Stable structures of oxidized carbon as in Earth's mantle



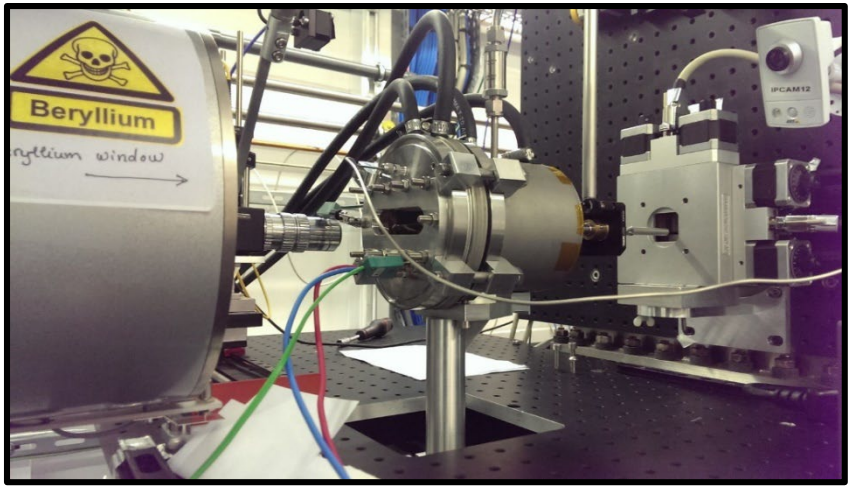
Source:
R. Dasgupta (2013)
Rev. Miner. Geochem.

In situ HP-HT experiments

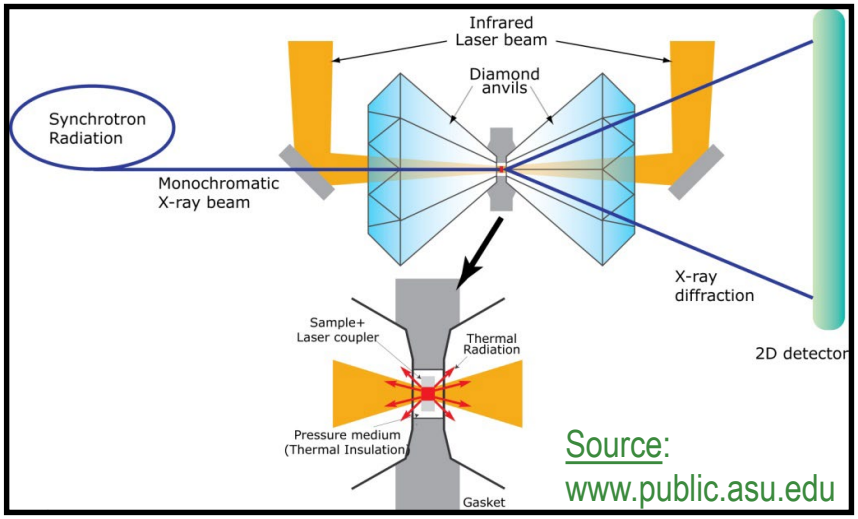


Tiny samples: 30 μm x 30 μm x 5 μm

External resistive-heating DAC in vacuum vessel
ALBA Synchrotron Light Source



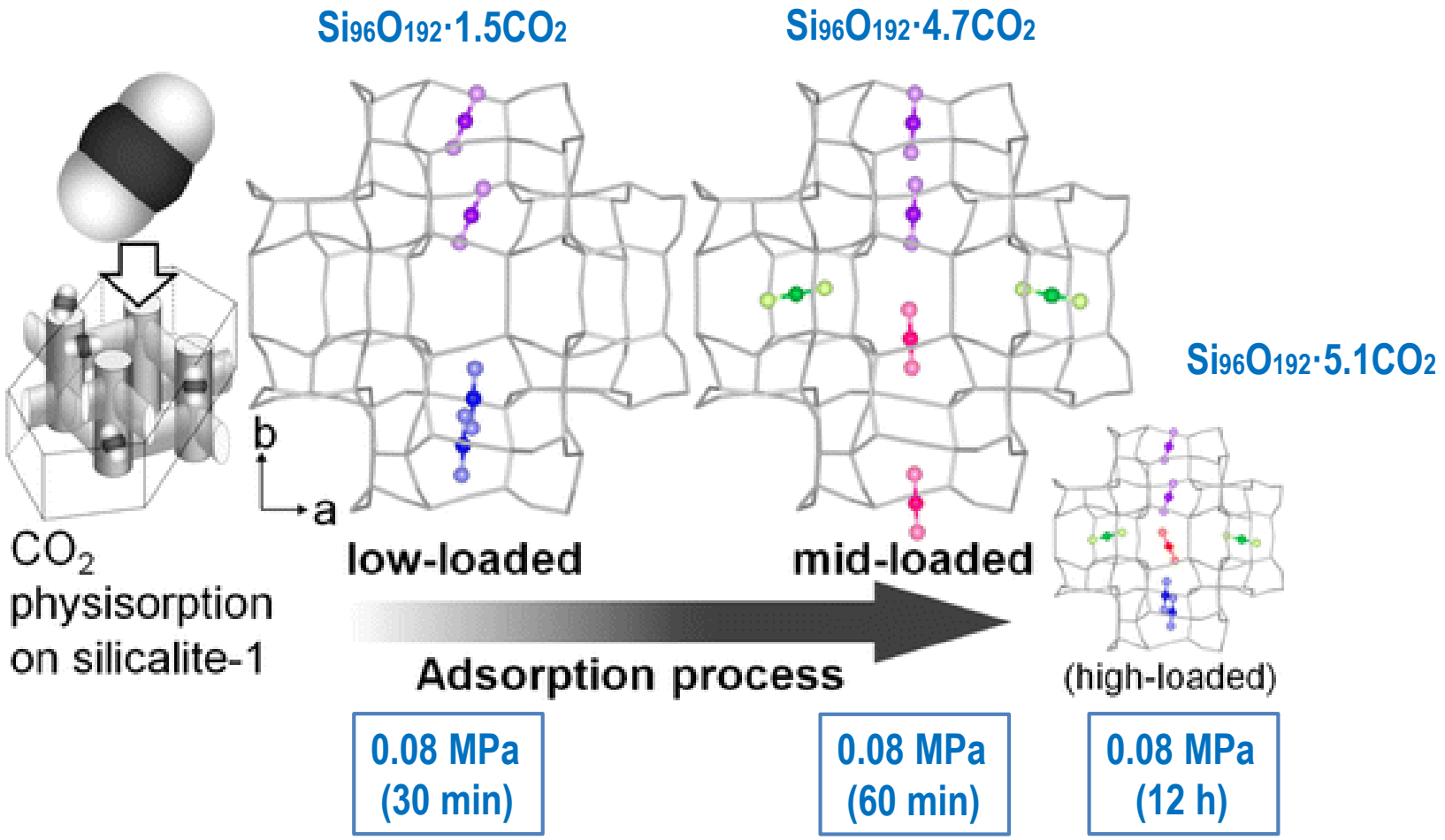
Double-sided laser-heating DAC
Advanced Photon Source



Pure-SiO_2 Silicalite-1 zeolite (S.G. *Pnma*)

Framework density:

17.9 T-atoms/1000 Å^3

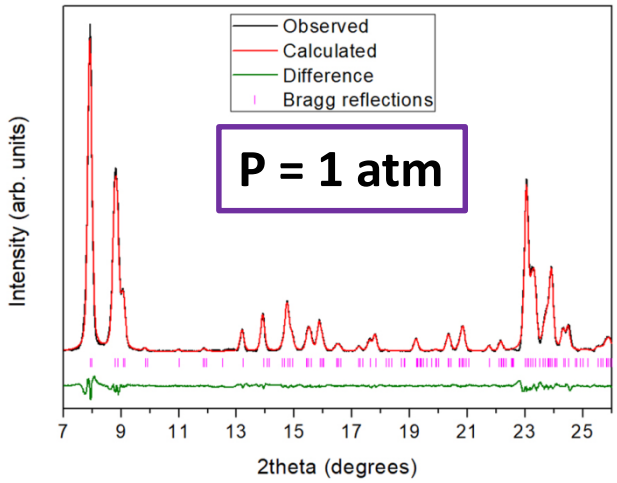


Langmuir Article pubs.acs.org/Langmuir

Adsorption Process of CO_2 on Silicalite-1 Zeolite Using Single-Crystal X-ray Method

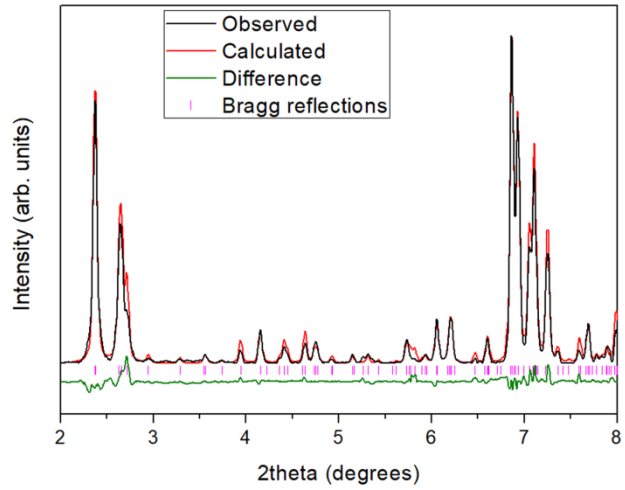
Shinjiro Fujiyama,* Natsumi Kamiya, Koji Nishi, and Yoshinobu Yokomori*

Langmuir 2014, 30, 3749−3753



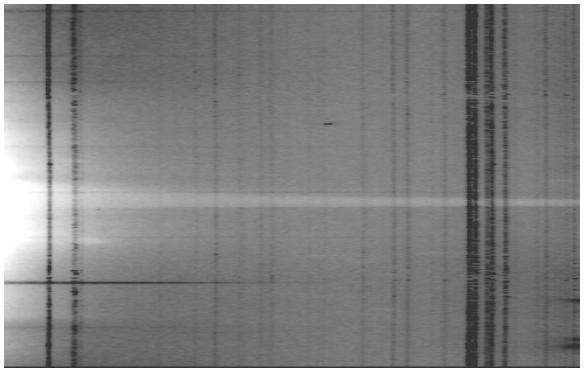
P = 1 atm

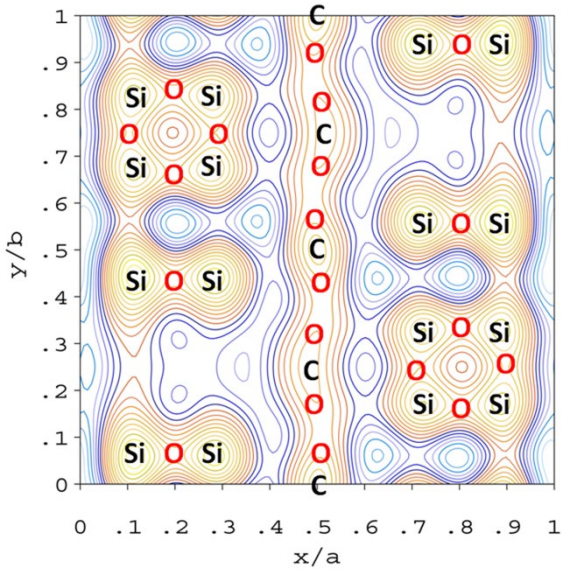
XRD pattern of empty Silicalite

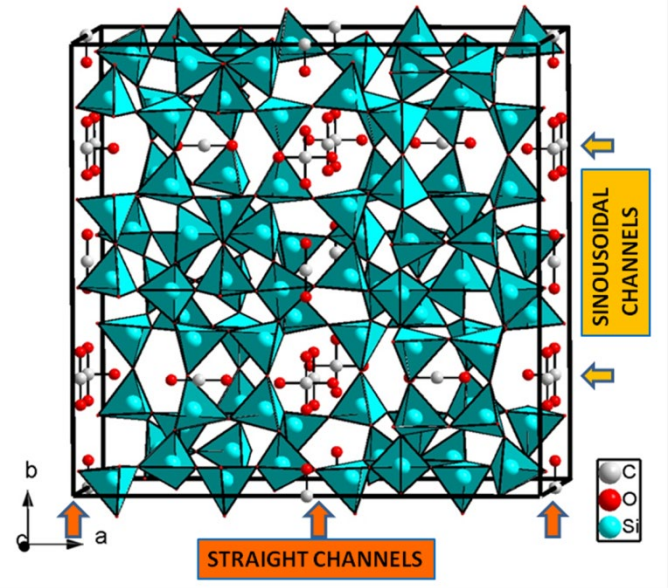


P = 0.5 GPa

Silicalite + CO2 in a DAC

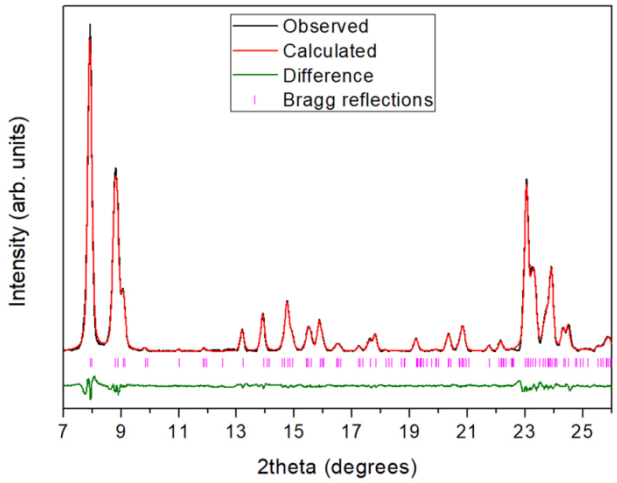




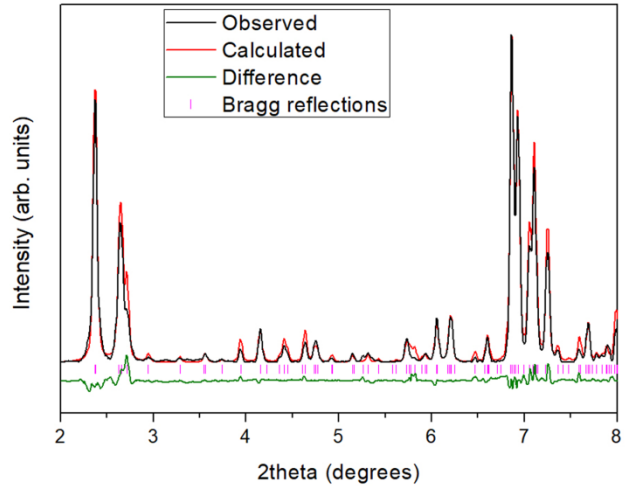


CO_2-filled Silicalite-1 zeolite ($Si_{96}O_{192}\cdot 16CO_2$)

Ratio CO_2/SiO_2 = 1/8

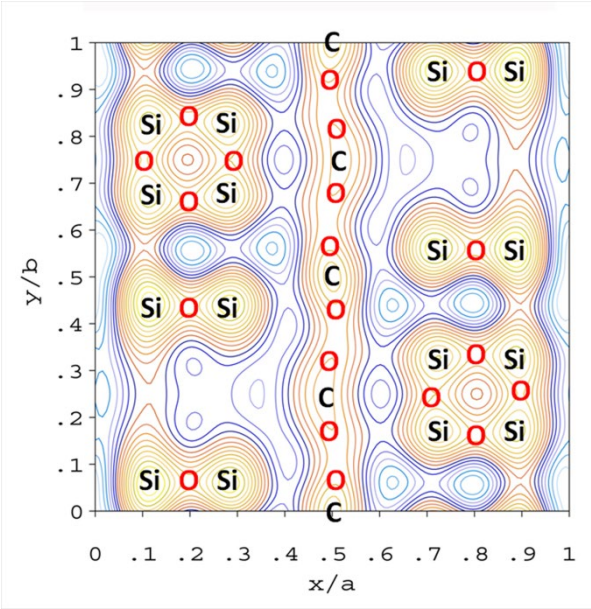


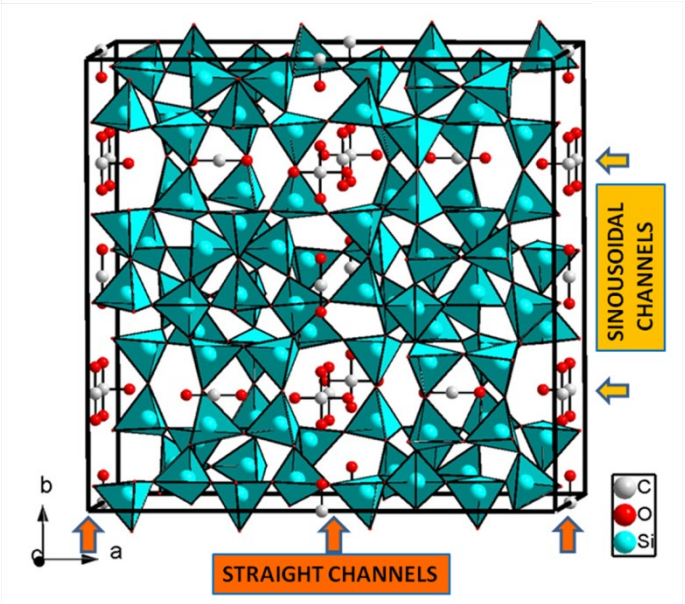
XRD pattern of empty Silicalite



P = 0.5 GPa

Silicalite + CO2 in a DAC





Inorganic Chemistry

Cite This: Inorg. Chem. 2018, 57, 6447−6455

An Ultrahigh CO_2-Loaded Silicalite-1 Zeolite: Structural Stability and Physical Properties at High Pressures and Temperatures

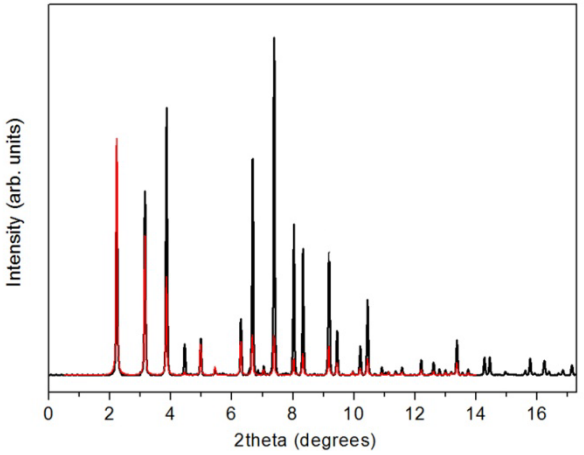
Tomas Marqueño,[†] David Santamaria-Perez,[†,*] Javier Ruiz-Fuertes,[†,‡] Raquel Chuliá-Jordán,[†] Jose L. Jordá,[§] Fernando Rey,[§] Chris McGuire,[∥] Abby Kavner,[∥] Simon MacLeod,[⊥,#] Dominik Daisenberger,[@] Catalin Popescu,[▽] Placida Rodriguez-Hernandez,[○] and Alfonso Muñoz[○]

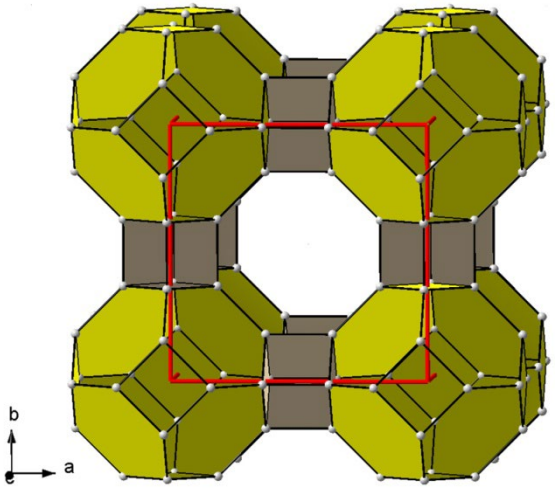
CO_2-filled Silicalite-1 zeolite ($Si_{96}O_{192}\cdot 16CO_2$)

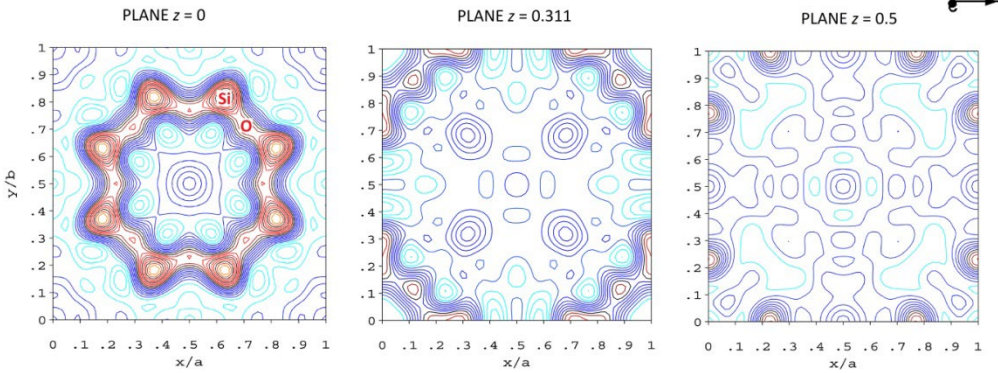
Pure-SiO_2 LTA zeolite (S.G. *Pm*-3*m*)

Framework density:

12.9 T-atoms/1000 Å^3



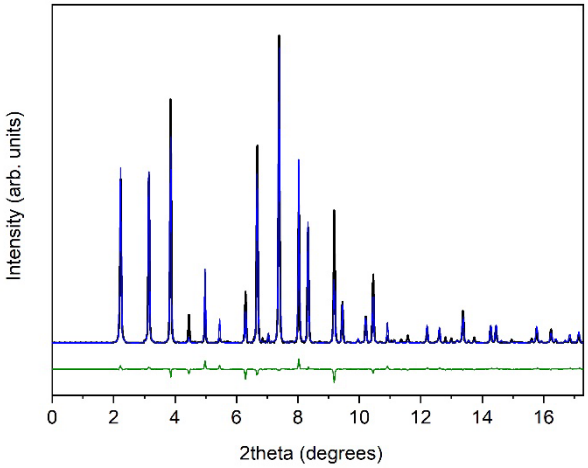


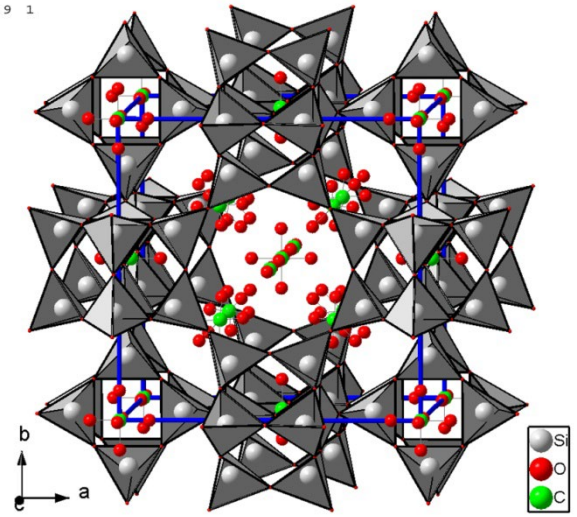


P = 0.4 GPa

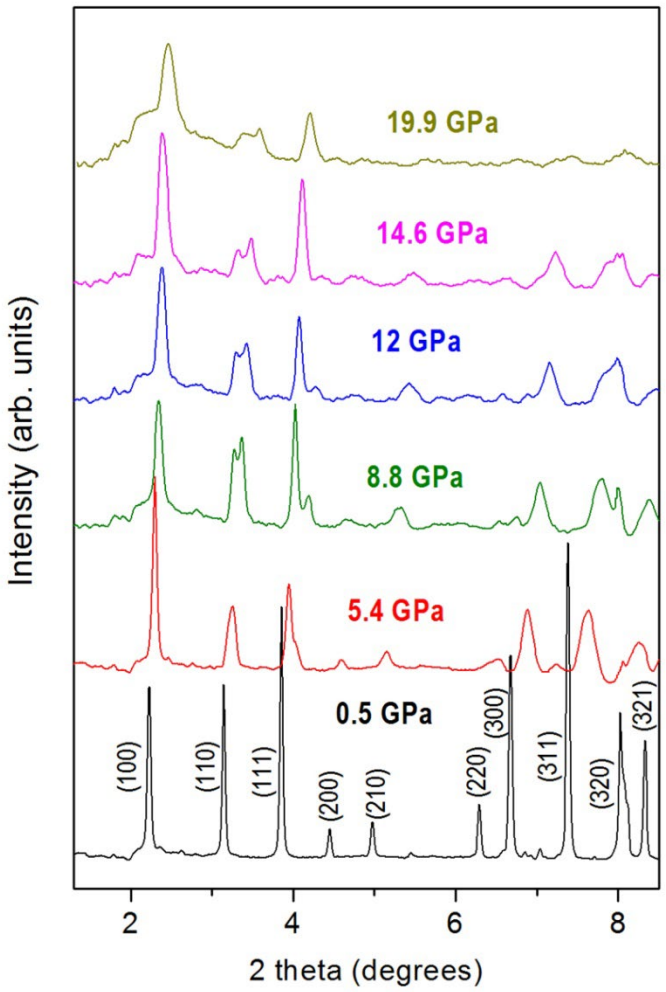
$Si_{24}O_{48}\cdot 13CO_2$

Ratio CO_2/SiO_2 = 13/24

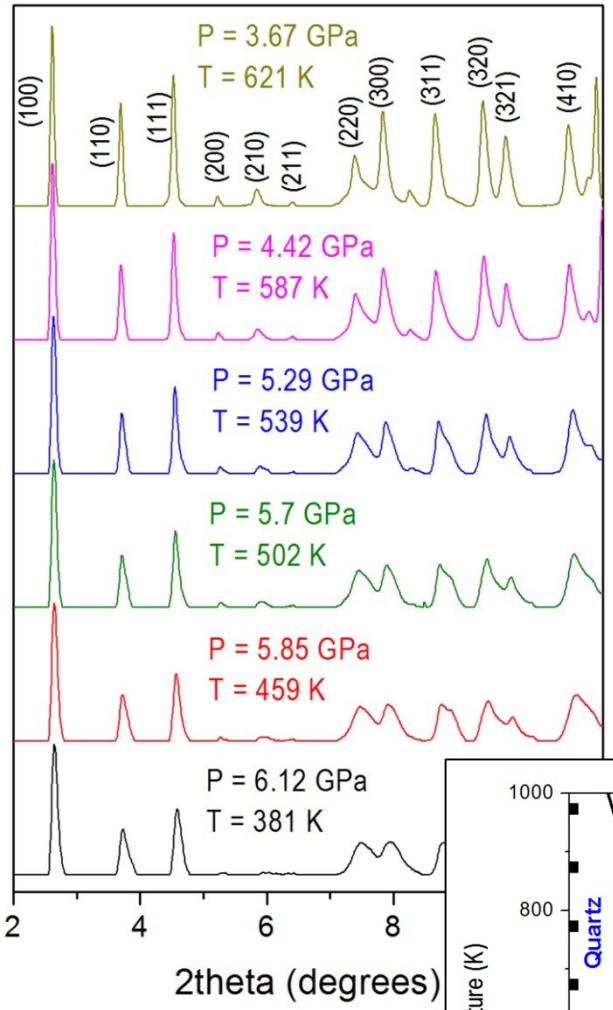




Ambient-temperature DAC



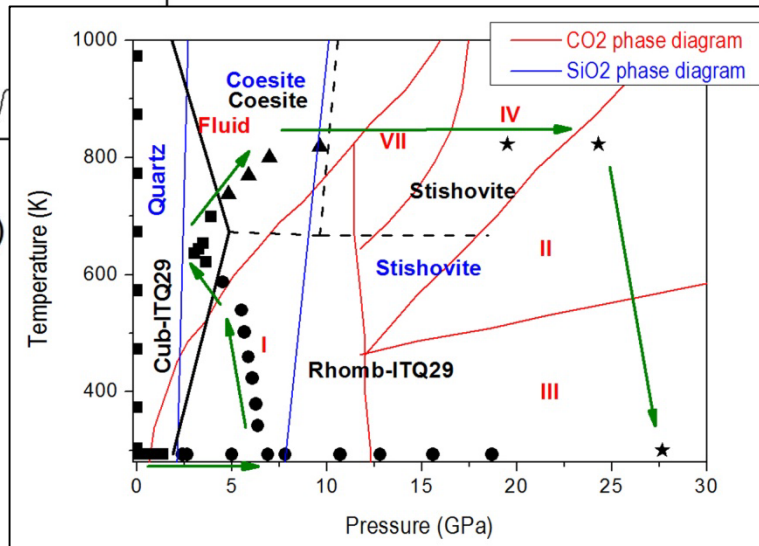
Resistive-heated DAC



- PIA Deactivation

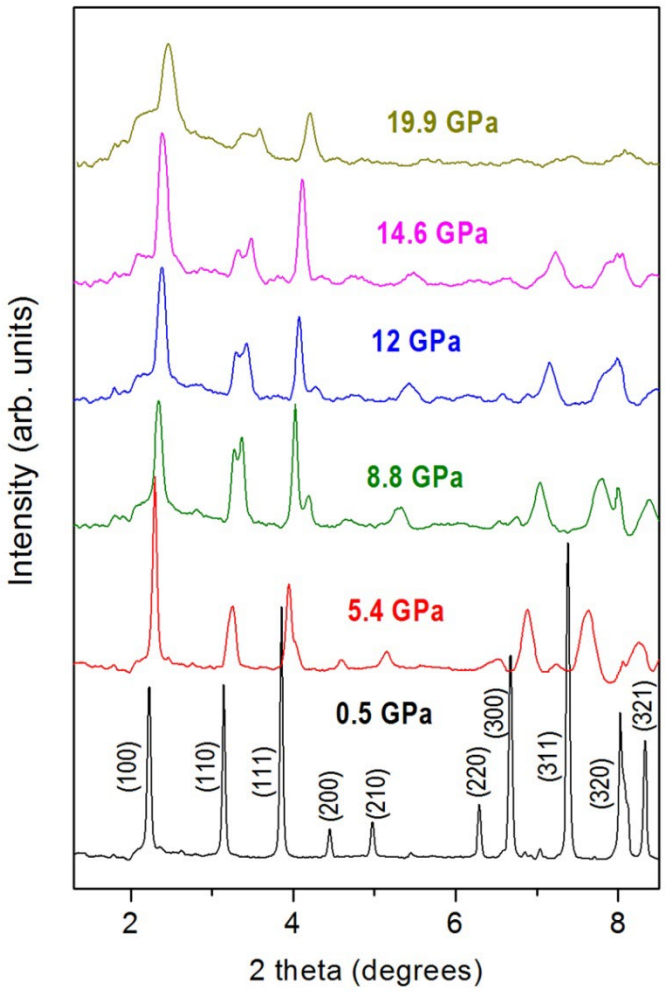
At
P = 4 GPa
T = 720 K

- Transformation into SiO_2 coesite + CO_2 liquid

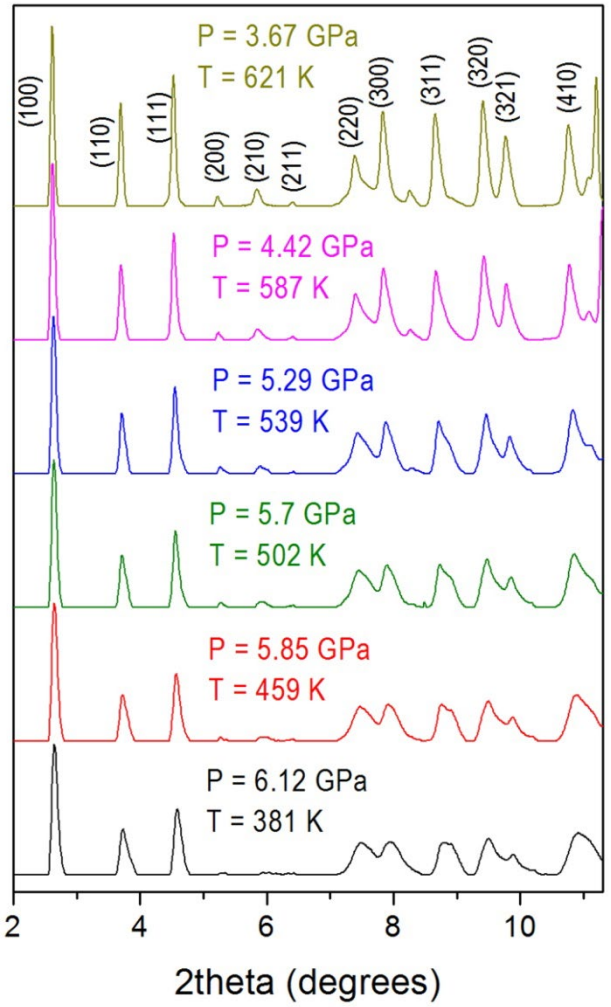


CO_2-loaded pure-SiO_2 LTA zeolite

Ambient-temperature DAC



Resistive-heated DAC



- PIA Deactivation

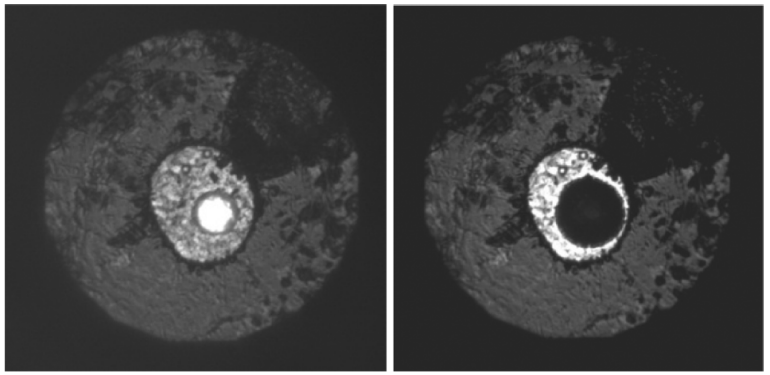
At
P = 4 GPa
T = 720 K

- Transformation into SiO_2 coesite + CO_2 liquid

CHEMISTRY OF MATERIALS

Structural Evolution of CO_2-Filled Pure Silica LTA Zeolite under High-Pressure High-Temperature Conditions

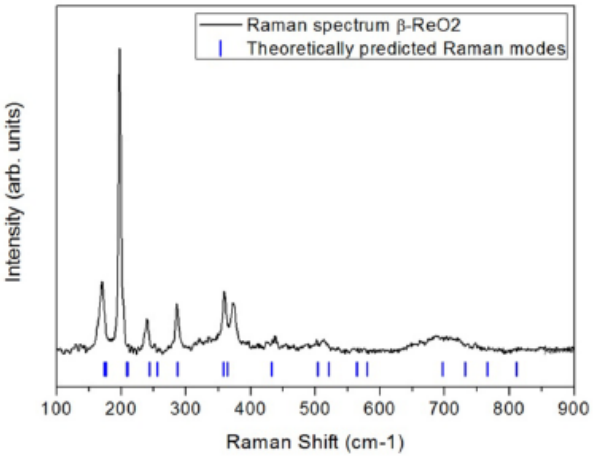
David Santamaría-Pérez,[*,†] Tomas Marqueño,[†] Simon MacLeod,[‡,§] Javier Ruiz-Fuertes,[†] Dominik Daisenberger,[⊥] Raquel Chuliá-Jordan,[†] Daniel Errandonea,[†] Jose Luis Jordá,[¶] Fernando Rey,[¶] Chris McGuire,[#] Adam Mahkluf,[#] Abby Kavner,[#] and Catalin Popescu[∥]



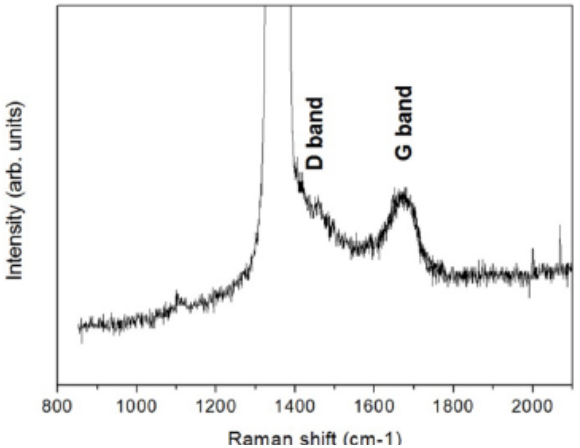
Laser-heated DAC

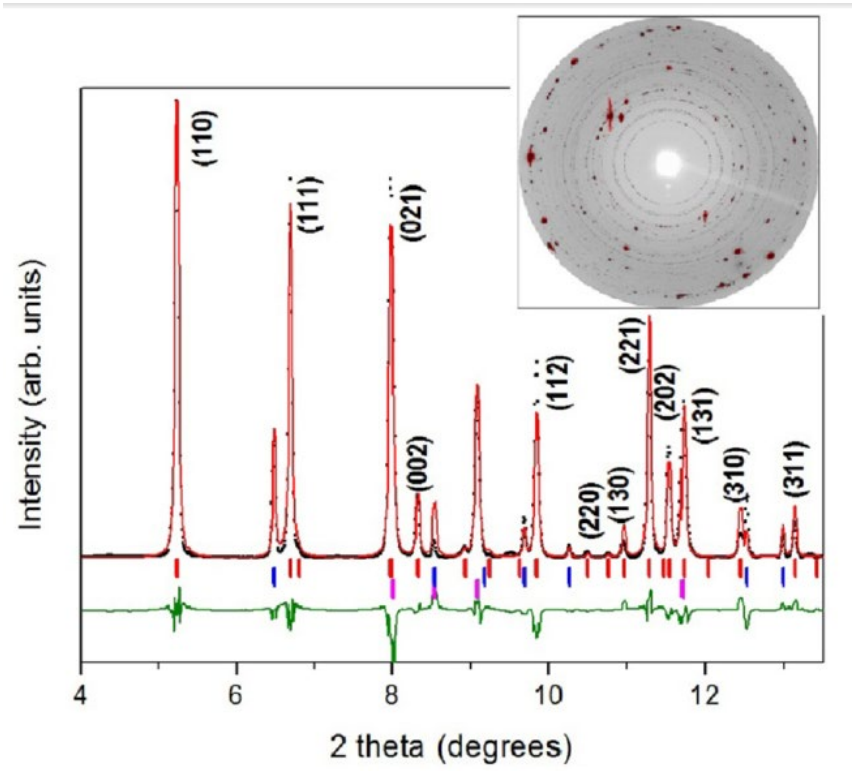
P > 2 GPa

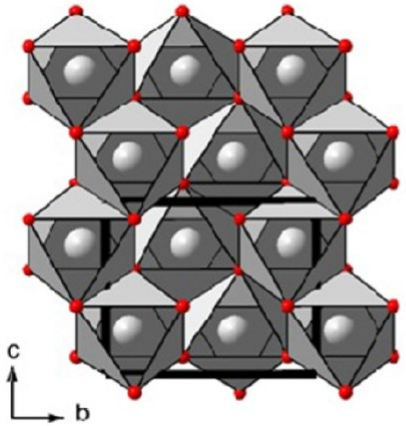
T > 1300 K

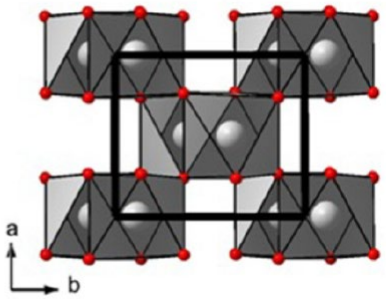


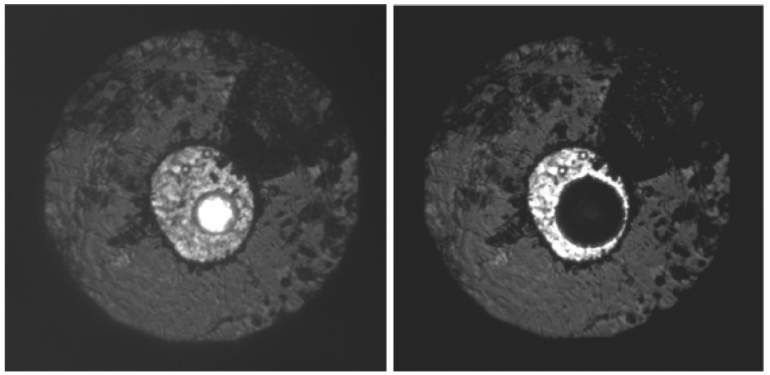
$SiO_2 + CO_2 + Re \rightarrow \beta\text{-}ReO_2 + C + SiO_2$



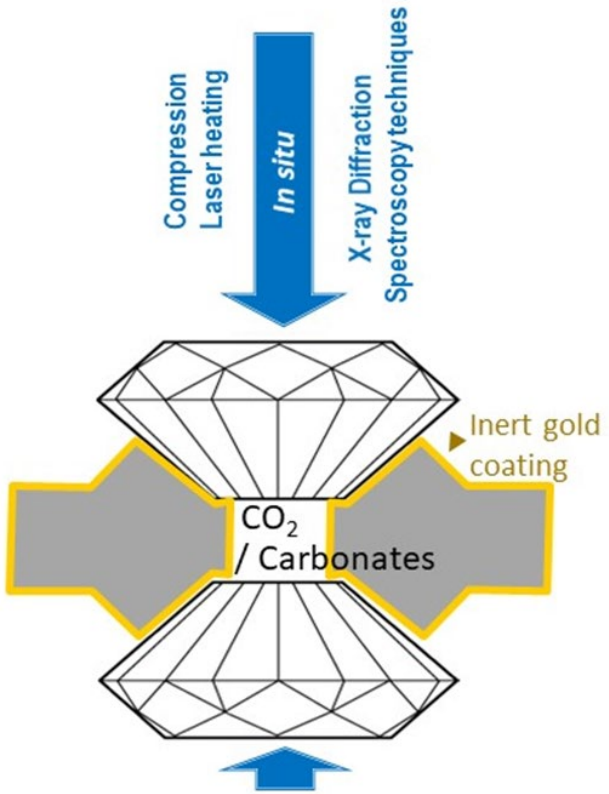




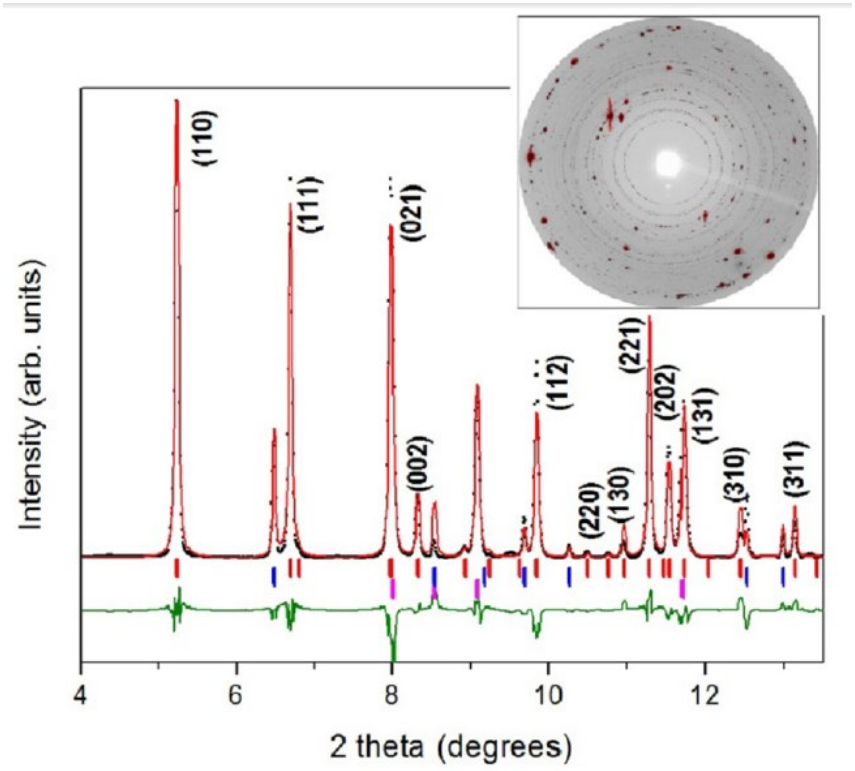




Laser-heated DAC



$$SiO_2 + CO_2 + Re \rightarrow \beta\text{-}ReO_2 + C + SiO_2$$



nature COMMUNICATIONS

Correspondence: Strongly-driven $Re + CO_2$ redox reaction at high-pressure and high-temperature

D. Santamaria-Perez[1,2], C. McGuire[1], A. Makhluf[1], A. Kavner[1], R. Chuliá-Jordan[2], J.L. Jorda[3], F. Rey[3], J. Pellicer-Porres[2], D. Martinez-García[2], P. Rodriguez-Hernández[4] & A. Muñoz[4]

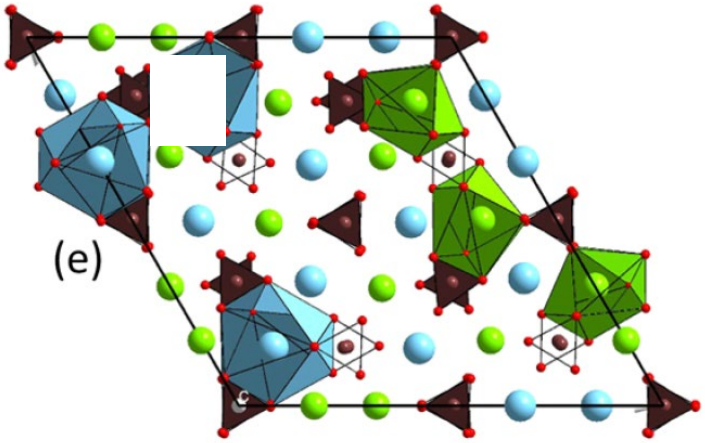
Nature Communications 7:13647

$BaO - CaO - CO_2$ System

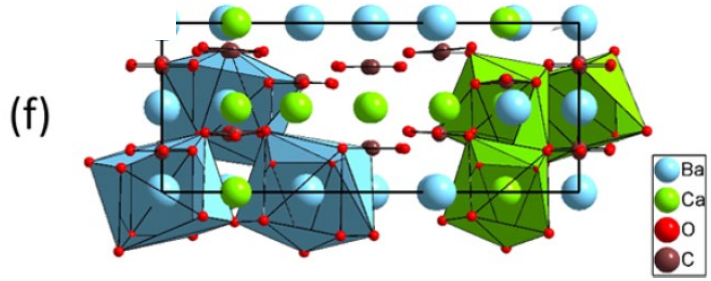
R. Chulia-Jordan, *et al.*
J. Earth Planet. Chem. 5, 1130 (2021).

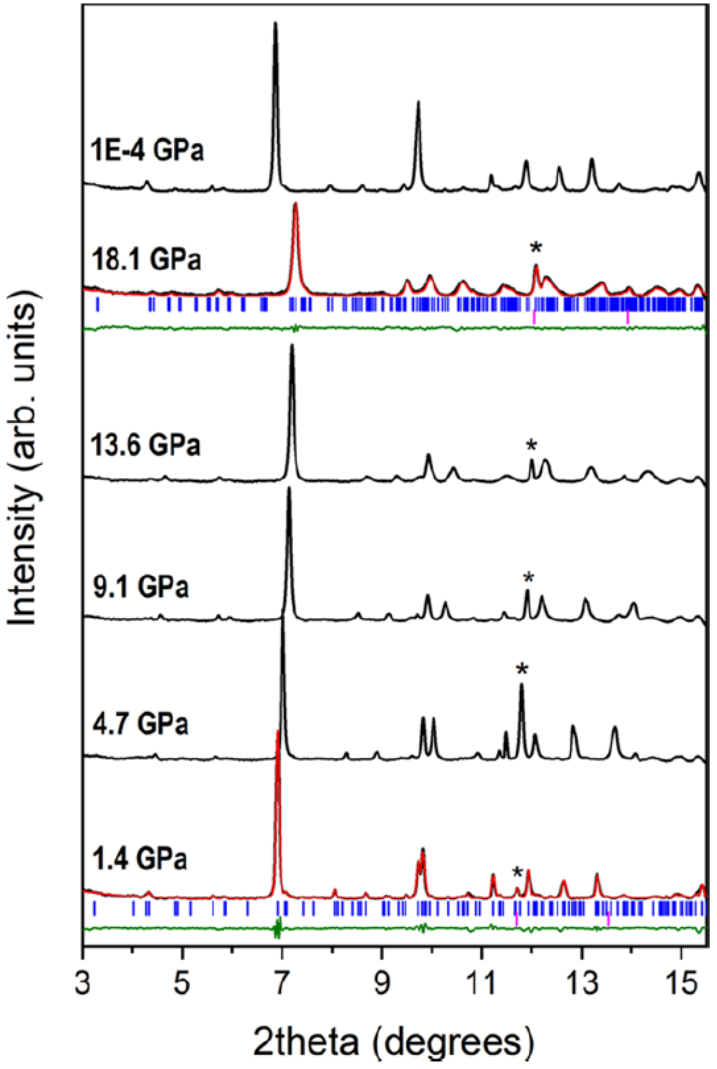
3 minerals in nature
$BaCa(CO_3)_2$

- Barytocalcite
- Paralstonite
- Alstonite



Alstonite - Determined by single-crystal XRD.

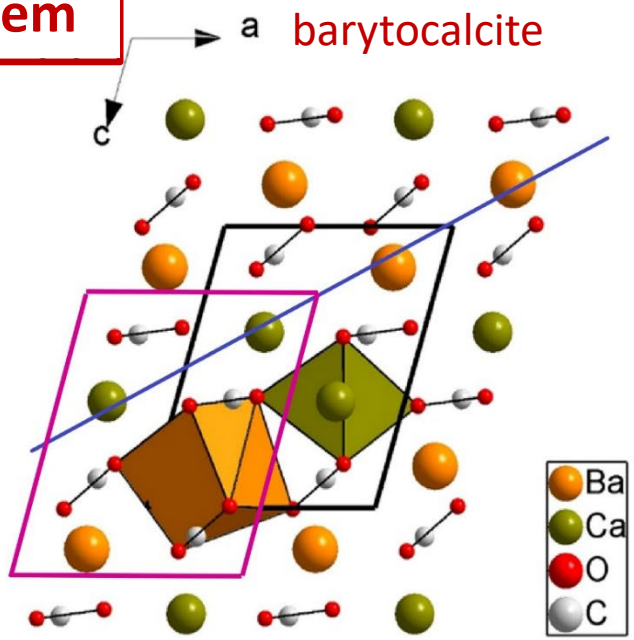


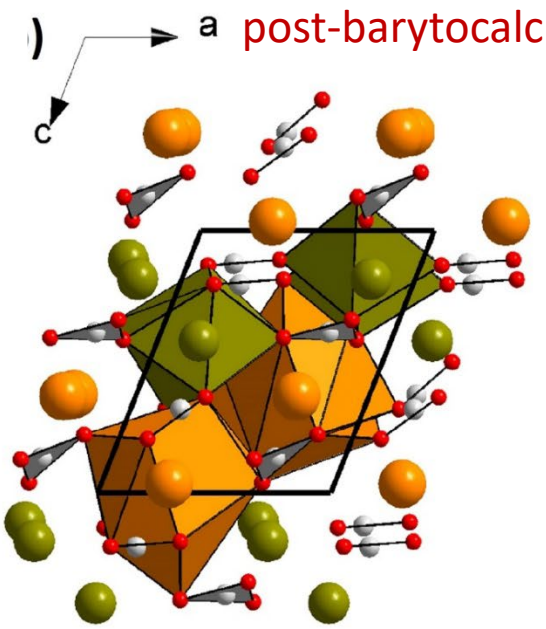


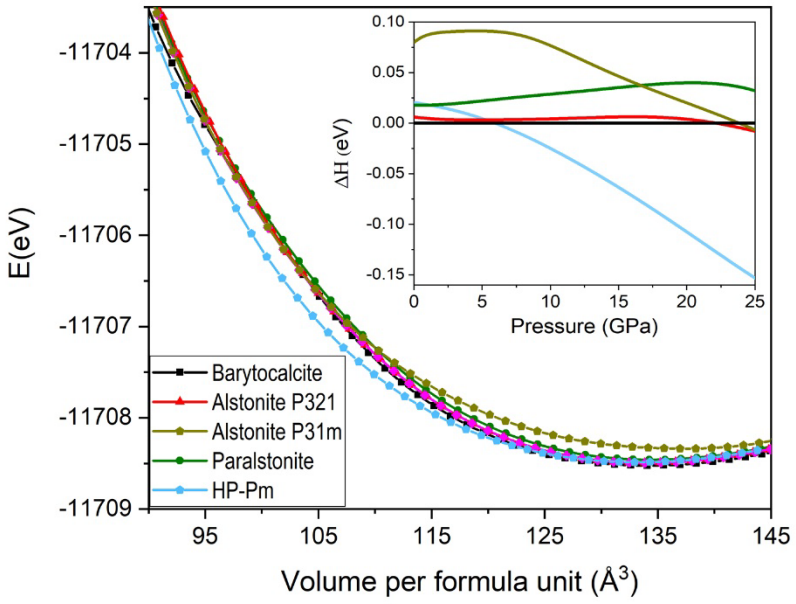
$BaO – CaO – CO_2$ System

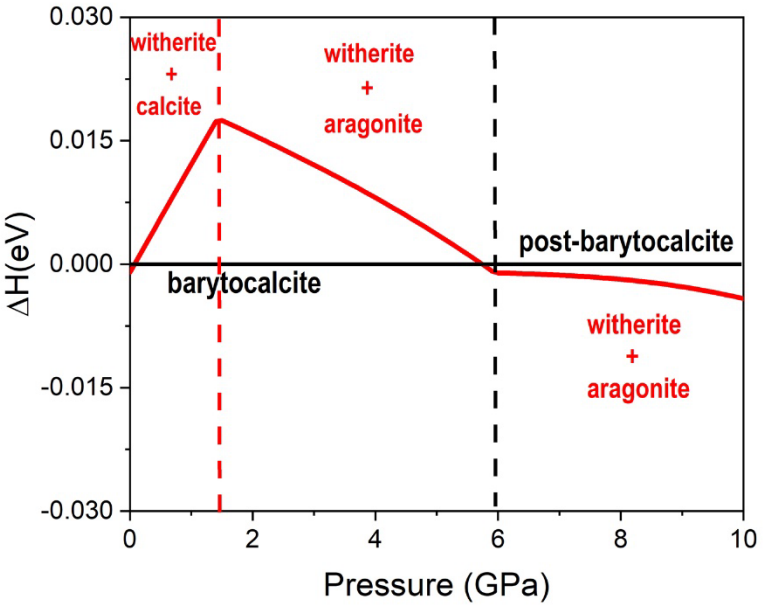
$BaCa(CO_3)_2$
post-barytocalcite

Chuliá-Jordán et al. (2022)
Sci. Rep. 12:7413



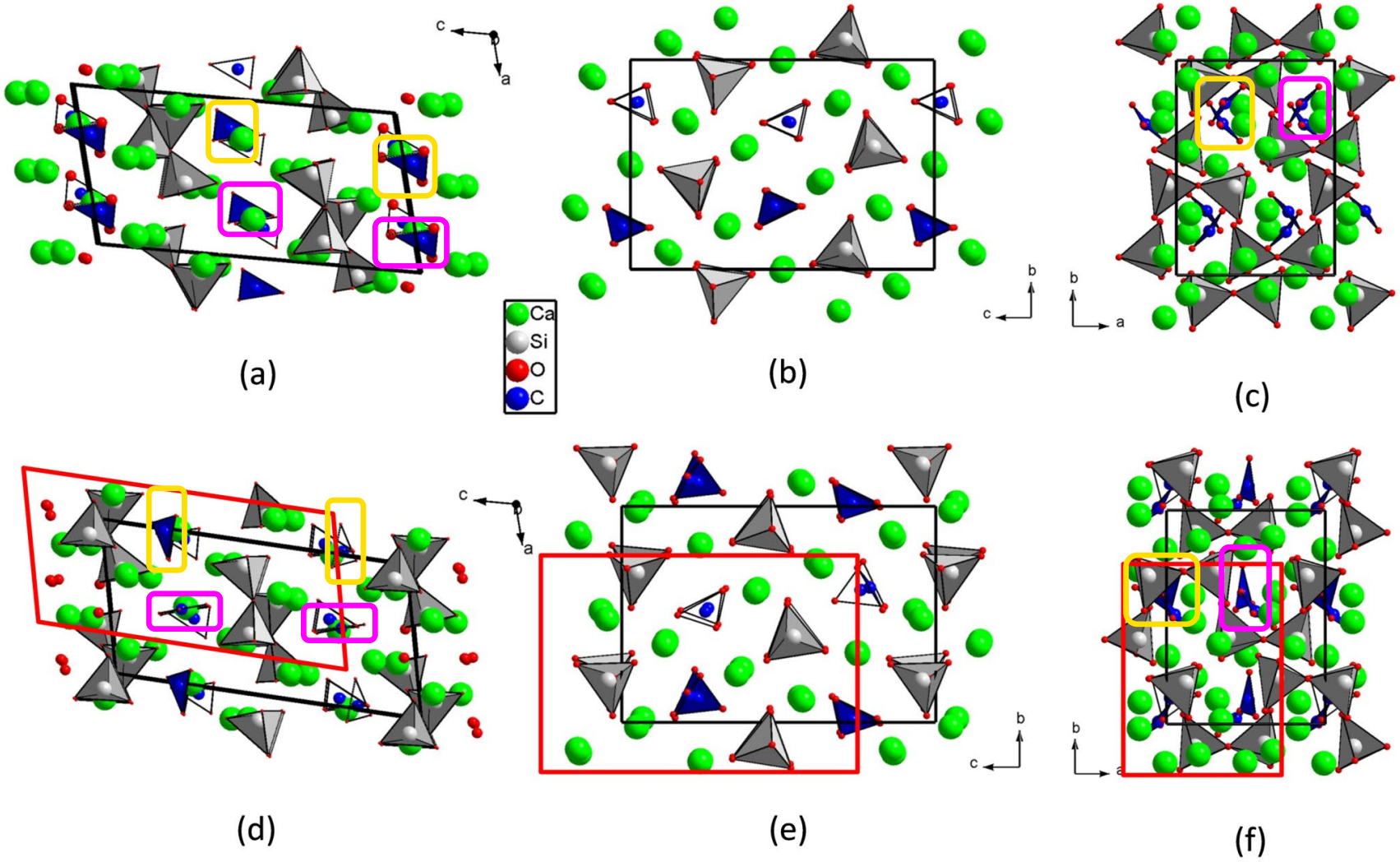






$CaO - SiO_2 - CO_2$ System

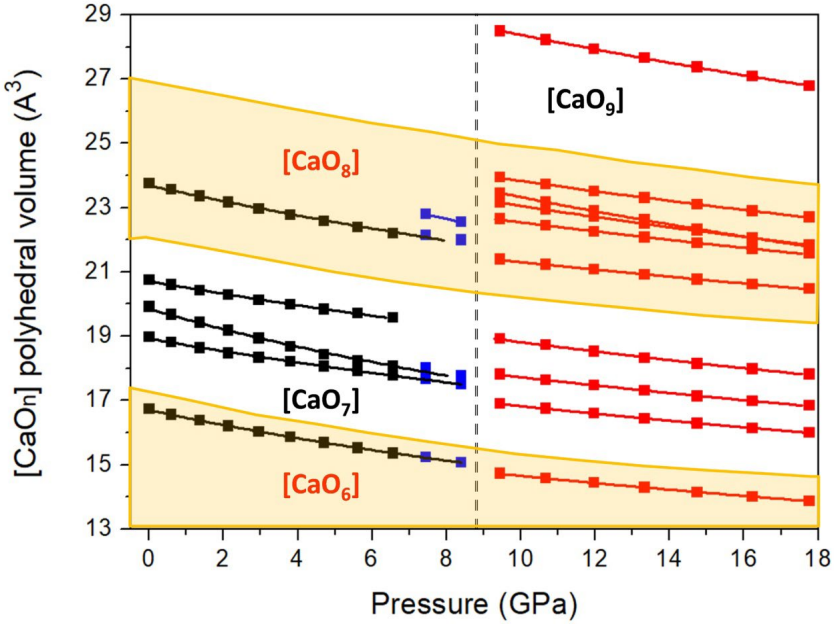
Novel dense silicate-carbonate phase: $Ca_5(Si_2O_7)(CO_3)_2$ post-tilleyite



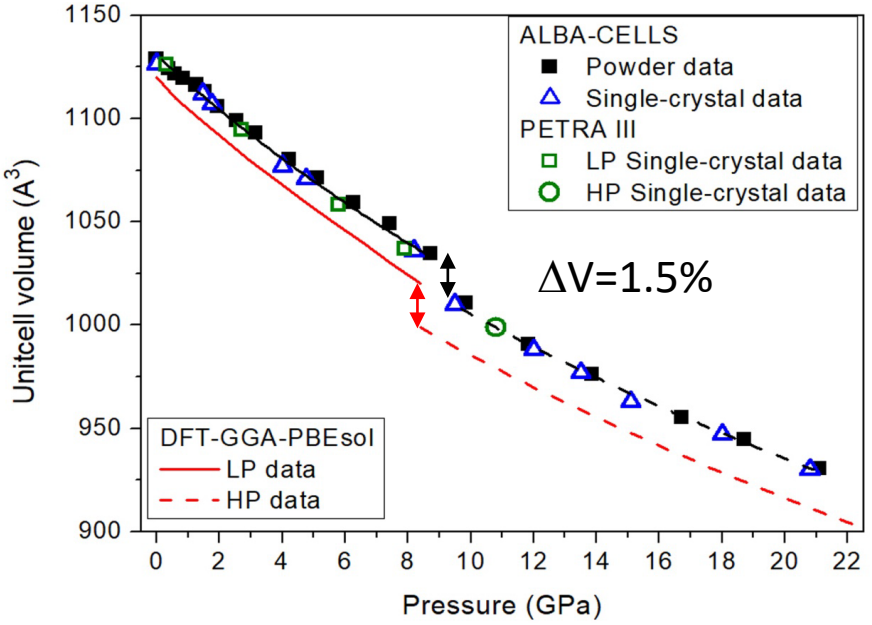
D. Santamaria-Perez, *et al*. Sci. Rep. 9, 7898 (2019)

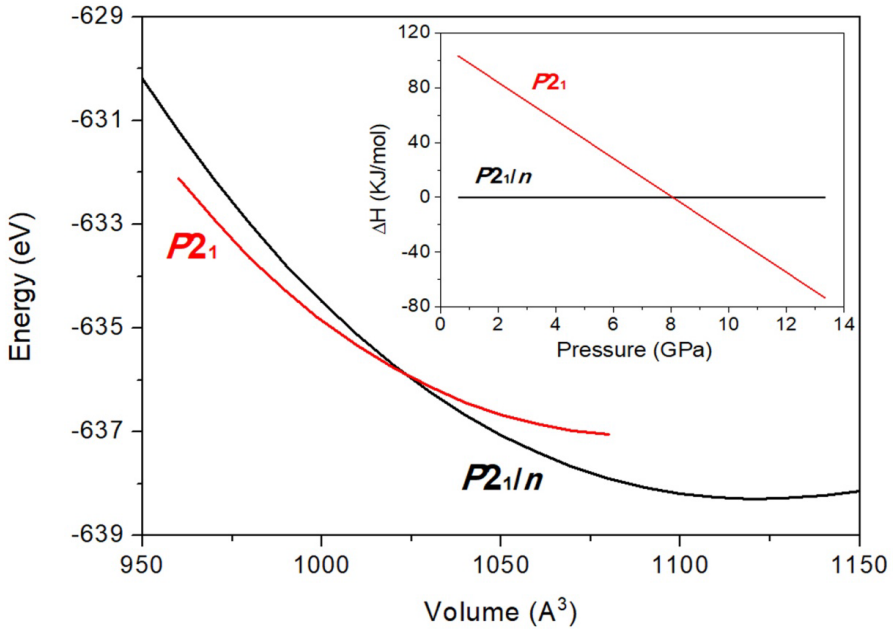
$CaO - SiO_2 - CO_2$ System

$Ca_5(Si_2O_7)(CO_3)_2$ post-tilleyite structural properties



Great diversity of coordination environments





D. Santamaria-Perez, et al. Sci. Rep. 9, 7898 (2019)

Summary – CO_2 and carbonates at HP-HT

- Combining good-quality powder XRD data, Rietveld refinement and difference-Fourier analysis is possible to accurately determine the content of adsorbed CO_2 and the location of molecules in zeolite frameworks.

- If compressed CO_2 is heated above 1300 K in the presence of rhenium or tungsten, these metals easily oxidize and carbon reduces to graphite and/or diamond.

- We have determined the P-T stability range of carbonate minerals and discovered several dense phases; e.g. alstonite, post-barytocalcite, post-tilleyite,

Still too much work to do
in CO_2-based systems at extreme conditions…

For more information:

https://extreme-carbonates.github.io/

MEMBERS

Dr. David Santamaría Pérez

Dr. Javier Ruiz Fuertes

Dr. Alberto Otero de la Roza

Dr. Raquel Chuliá Jordán

ALBA

Dr. Catalin Popescu