

The critical role of stored energy in metamorphism

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The mechanical behaviour of the Earth is widely assumed to change with increasing timescale as rocks progressively relax imposed stresses [1]. On short timescales, elasticity dominates and its release drives earthquakes [2]; at intermediate timescales, viscoelastic relaxation facilitates processes such as glacial rebound [3]; whereas over geological timescales viscous deformation dominates, allowing the mantle to flow [4]. Energetically, this progression implies that mechanically stored potential energy becomes increasingly irrelevant to long-term geological processes such as metamorphism. Here we challenge this assumption using time-resolved deformation experiments that isolate changes in stored energy during metamorphic reaction. We show that increasing stored energy causes reaction to occur sooner and faster, increases nucleation density and reduces product grain size. Comparison with experiments from crustal and mantle materials shows that this influence persists across different reactions and deformation regimes. These results demonstrate that stored energy can exert a first-order control on geological transformation despite its comparatively small contribution to the total energy budget. Because stress and stored energy are heterogeneous across materials and scales, our results place stored energy alongside water as a driver of metamorphic change and provide experimental support for a view of the Earth in which changes in Helmholtz energy influence the emergence of material instabilities [5–8].

Metamorphism and deformation are two fundamental sources of transformation in geology that operate simultaneously deep within the Earth. It has long been known that they interact, but there remains no agreement about which aspect of the mechanical state is relevant to metamorphism [9,10]. Classically, deformation is understood to influence metamorphism by changing the physical environment in which reaction occurs: generating defects and new interfaces, reducing grain size, and modifying pathways for fluid and chemical transport [11–13]. In parallel, a long-running thermodynamic debate has centred

51 on which stress state describes pressure (P) under non-hydrostatic conditions [10, 14–17],
52 and consequently how stress changes the Gibbs free energy (G),

$$G = U + PV - Ts$$

53 where U is the total internal energy of the system, V its volume, T temperature and s
54 entropy. This pressure-centred description, however, obscures one part of the mechanical
55 energy budget: the energy stored within the stressed material itself. A useful alternative
56 formulation separates the pressure-volume contribution from the Helmholtz free energy
57 (ψ),

$$\psi = U - Ts$$

58 where

$$G = \psi + PV$$

59 Deformation can change ψ by storing energy in recoverable elastic strain and in
60 microstructural defects such as dislocations [13, 18–21]. This distinction provides a
61 different way of asking how deformation influences metamorphism. Rather than asking
62 only which stress should define pressure, we can ask whether changes in the energy stored
63 by a deforming material influence its transformation. The common assumption in geology
64 is that this contribution can be neglected over geological timescales because it is small
65 relative to other energetic contributions [22] and the stresses maintaining it relax much
66 faster than geological processes occur [8, 23, 24].

67

68 This assumption sits uneasily with both observations and theory. Elastic strains and
69 “residual stresses” are routinely measured in exhumed metamorphic rocks, where they
70 can remain preserved over geological timescales [25, 26]. Indeed, elastic thermobarometry
71 explicitly exploits this preservation, using residual strains in mineral inclusions to recon-
72 struct the conditions at which minerals were entrapped [25]. More fundamentally, the
73 Earth is not a mechanical system that is stressed once and then allowed to relax. During

74 tectonic deformation, stored energy is continuously generated, redistributed and dissipated
75 as rocks respond to evolving stresses, material properties and boundary conditions [7].
76 Stress relaxation therefore does not necessarily imply the disappearance of stored energy
77 from the system. Consistent with this view, theoretical descriptions of coupled Earth
78 dynamics predict that even modest variations in stored energy can remain relevant over
79 geological timescales [27], govern the spontaneous emergence of tectonic localisation [7, 28],
80 and regulate the long-term strength of the lithosphere [8]. The difference between these
81 views is subtle but consequential. In one, stored energy becomes dynamically irrelevant as
82 stresses relax; in the other, it is an evolving component of the energy available to drive ge-
83 ological change. Determining whether stored energy can influence material transformation
84 therefore tests a fundamental assumption about the energetics of the long-term dynamics
85 of the Earth.

86 **Time-resolved deformation experiments**

87 We test whether changes in stored energy exert a first-order control on the relationship
88 between tectonic deformation and metamorphic transformation using synchrotron-based
89 time-resolved deformation experiments in the X-ray-transparent miniature triaxial rock
90 deformation apparatus Mjöltnir [29]. This allows us to control the mechanical state of
91 a rock while simultaneously measuring the evolution of reactants and products during
92 reaction.

93
94 Our experiments use the dehydration of gypsum, a well-established model system for
95 investigating the dynamics of crustal metamorphism [30–35]. Natural Volterra alabaster
96 samples were reacted at constant temperature (125 °C), confining pressure (20 MPa) and
97 pore-fluid pressures of 1–5 MPa. Gypsum reacts to form bassanite and water, accompanied
98 by a ~30% reduction in solid volume. We compare a hydrostatic reference experiment, in
99 which the principal stresses are equal, with experiments subjected to progressively larger
100 differential stresses.

The maximum differential stress imposed on the unreacted samples remained below the yield stress of gypsum in every experiment. The samples therefore accumulated additional elastic strain energy without undergoing macroscopic irreversible deformation before reaction. Across the experimental series, the thermal, pressure-volume and reaction contributions to the energy budget were effectively constant, such that the systematically varied contribution was the elastic strain energy stored in the sample. Relative to the hydrostatic reference, we impose a maximum increase in stored elastic energy density of $1.08 \times 10^4 \text{ J m}^{-3}$, calculated from $u = \sigma_{\text{diff}}^2 / (2E)$. This is only 0.06% of the chemical driving force of $1.70 \times 10^7 \text{ J m}^{-3}$ associated with the 24.4°C thermal overstep of all experiments run at 125°C [36] (cf. Supplemental materials). This experimental design isolates the effect of changing stored mechanical energy on the progress of a metamorphic reaction.

At the conditions of all experiments, gypsum dehydration is thermodynamically permitted. We therefore do not test whether stored energy shifts the equilibrium conditions of the reaction, but whether changing this energetic contribution affects its kinetics. Our experiments extend from hydrostatic conditions to immediately below the elastic limit of gypsum, bridging a gap between time-resolved measurements of reaction products at low differential stress [37, 38] and other kinetic experiments involving substantial irreversible deformation [13, 39]. Together, these experiments allow us to isolate the effect of stored elastic energy before irreversible deformation and its associated dissipation become dominant.

Time resolved in situ data were collected at the TOMCAT beamline of the Swiss Light Source (SLS), the PSICHE beamline of Synchrotron SOLEIL, and the I13-2 beamline of Diamond Light Source (DLS) for X-ray microtomography (μCT), and at the I15 beamline of Diamond Light Source for high-energy X-ray diffraction.

The effect of stress on reaction kinetics

Increasing differential stress systematically accelerates gypsum dehydration (Figure 1). When the evolution of bassanite molar fraction X with time is fitted using an Avrami kinetic law,

$$X(t) = 1 - \exp \left[- \left(\frac{t - t_0}{\tau} \right)^n \right],$$

clear trends are observed in the induction time t_0 , characteristic time τ , rate constant ($k = \tau^{-n}$) and maximum reaction rate. With increasing differential stress the reaction is observed to happen up to ~ 5 times sooner (Figure 2a) and with ~ 4 times faster maximum reaction rates (Figure 2b). The Avrami exponent remains approximately constant across the experiments ($n_{\text{avg}} = 2$, Figure 2c) and is consistent with the predominantly 1D growth of the observed bassanite needles. Microstructural analysis shows that a large amount of nucleation occurs in the first half of the experiment, but it is unclear if continuous or true burst nucleation is occurring. When comparing the microstructures of three experiments at the same reaction extent it is observed that as differential stress increases there is an increase in the number of the smallest bassanite crystals ($< 1000 \mu\text{m}^3$) and the average bassanite crystal size decreases (Figure 1a and 2d). We interpret differential stress to most strongly impact the nucleation phase of the reaction by changing the onset time and the nucleation density, with changes in growth probably being reflected in the change of the maximum reaction rate. These results demonstrate that stored energy has a significant impact on the geological process of metamorphism.

To understand how far reaching this impact is, we compare gypsum dehydration with experiments on reactions in lower-crustal and upper-mantle materials. We focus on the change in characteristic reaction time ($X = 1 - e^{-1} \approx 63\%$ reacted) between hydrostatic and differentially stressed experiments. Using the change relative to a hydrostatic reference allows comparison between experiments conducted at different temperature, pressure and pore-fluid pressure conditions. Experimentally measured peak differential stresses

are normalised by each material's yield stress so that materials can be compared. Both quantities are important: yield defines the transition from predominantly elastic energy storage to increasingly dissipative deformation with energy stored in lattice strains around deformation induced defects, whereas peak stress records the maximum stress sustained by the material before weakening and therefore the maximum stress available to contribute to stored energy. Figure 3 therefore provides a material- and condition-independent view of the experimental record of the effect of differential stress on reaction kinetics in the lithosphere. Figure 3 clearly shows that most of the hastening of reaction occurs as differential stress increases below yield, although deformation above yield continues to accelerate reaction.

To understand this pattern quantitatively, we estimate the stored mechanical energy accumulated in each experiment. We consider two contributions: recoverable elastic strain energy stored during loading and the energy retained around crystal defects known as dislocations generated during post-yield deformation. The stored energy density (u_{total}) is therefore approximated as

$$u_{total} = u_{elastic} + u_{dislocation} \quad (1)$$

$$u_{elastic} = \frac{\sigma_{diff}^2}{2E} \quad (2)$$

$$u_{dislocation} = \frac{1}{2}\mu b^2 \rho, \quad \rho = \left(\frac{\sigma_{diff}}{\alpha \mu b}\right)^2 \quad (3)$$

Where σ_{diff} , E , μ , b , ρ , α are the differential stress, Youngs modulus, Shear Modulus, burgus vector, dislocation density and an experimentally defined constant [40].

Despite the large differences in materials and experimental conditions, reaction kinetics are similarly sensitive to deformation even though the estimated changes in stored energy span more than two orders of magnitude (Figure 4). This variation in absolute stored energy

reflects differences in the elastic and crystal-plastic properties of the materials, as described by Eqs. 2 and 3, rather than differences in the underlying energy-storage mechanisms. Figure 4 shows that approximately 75% of the stored energy is accumulated elastically before yield, with the remaining $\sim 25\%$ retained in crystal defects generated during post-yield deformation. This partitioning provides a first-order explanation for the trend in Figure 3: most of the acceleration of reaction occurs below yield, where elastic energy is accumulated, while the continued addition of stored energy in crystal defects provides a mechanism for the smaller additional acceleration above yield. Together, these experiments outline that changes in stored energy hold the key to a common energetic description of the effect of differential stress on reaction kinetics across different materials and reactions.

Why stored energy matters in geodynamics

The Earth is heterogeneously stressed at all scales, and our results show that the resulting variations in stored energy can produce large differences in metamorphic reaction kinetics. This returns us to the assumption with which we began. For gypsum, the maximum variation in stored mechanical energy is only $\sim 0.06\%$ of the thermodynamic driving force associated with the 24.4°C experimental thermal overstep, yet this small energetic perturbation causes reaction products to appear up to $\sim 80\%$ sooner. The energetic landscape governing metamorphism is therefore not defined solely by pressure, temperature and composition, but is also modified by the local mechanical state.

This provides a different way to interpret spatially heterogeneous metamorphic reaction. Variations in reaction extent are commonly attributed to local kinetic barriers that prevent or delay reaction from occurring [41–43]. Our results show that this need not be the case. Two neighbouring rock volumes experiencing similar bulk P-T-X conditions may transform at markedly different rates simply because their stored-energy states differ. Regions preserving metastable reactants may therefore not have encountered an additional local barrier; they may simply have transformed more slowly over the duration

of a geological event. Stored-energy heterogeneity can thus be translated directly into reaction heterogeneity, even without large irreversible strains. Just as the heterogeneous distribution of water can determine where and how rapidly metamorphic reactions proceed, variations in stored energy can generate large differences in reaction rate. Unlike water, however, these variations arise directly from the mechanical state of the rock, providing an intrinsic coupling between deformation and metamorphic transformation.

Extending this further beyond the immediate consequences of our experiments, variations in stored energy in space and time might more faithfully describe the location, character and initiation of localisation phenomena in nature, including boudinage, folding and shear-zone formation [7, 28, 44]. At present, many geodynamic simulations of the lithosphere use prescribed seeds that mimic the inevitable geometrical and material complexity of rocks to initiate processes and structures. This is needed in part because these models do not explicitly represent solid mechanics and the associated storage and redistribution of mechanical energy. Our results suggest that such imposed heterogeneity may not always be necessary: heterogeneities in elastic material properties and energy-storing microprocesses will naturally perturb the potential-energy field, and these perturbations can influence where subsequent transformation and dissipative processes occur.

Our experiments show that changes in stored energy can influence material transformation before large irreversible strains develop, while our comparison with previous experiments indicates that stored energy continues to contribute after yield. The interaction between energy storage, transformation and dissipation can therefore persist across deformation regimes. This provides experimental support for the modelling paradigm in which changes in Helmholtz energy contribute to the emergence of transformations and localising instabilities in rocks [5–7].

When stored energy matters in geodynamics

Whether stored mechanical energy influences a geological transformation should therefore depend not simply on the absolute duration of the geological process, but on the relative timescales of mechanical relaxation and chemical transformation. The characteristic time for viscous relaxation of stored elastic stress is given by the Maxwell time, $\tau_M = \eta/\mu$, where η is viscosity and μ is the shear modulus. Assuming that the rate-limiting chemical process is diffusional, its characteristic timescale is $\tau_D \sim L^2/D$, where L is the characteristic diffusion length and D is the chemical diffusivity [7]. Consequently, chemical transformation can respond to a heterogeneous stored-energy field wherever $\tau_D \lesssim \tau_M$, even where the material behaves viscously over longer length and time scales. Equating these timescales defines a characteristic length scale,

$$L_c \sim \sqrt{\frac{D\eta}{\mu}},$$

below which chemical diffusion can operate before mechanical relaxation. Because τ_D decreases with the square of length scale, this condition becomes increasingly favourable towards the grain and sub-grain scales over which metamorphic reactions operate. A rock may therefore behave viscously at the macroscopic scale while retaining stored mechanical energy sufficiently long for local chemical transformation to respond to it. The relevance of stored energy to metamorphism is consequently not determined simply by whether rocks are “elastic” or “viscous” on geological timescales, but by the competition between chemical transformation and mechanical relaxation at the length scale of the reaction.

Outlook

Our results show that predicting geological transformation requires consideration not only of pressure, temperature and composition, but also of the evolving mechanical state of the material. This is particularly important because rates, rather than equilibrium states alone, govern the evolution of many coupled thermo-hydro-mechanical-chemical systems [45]. Yet current formulations generally do not capture the feedback demonstrated here, in which

changes in stored mechanical energy modify reaction rates and transformation in turn modifies the mechanical state. Quantifying how stored energy is generated, redistributed and dissipated, and incorporating its effect on reaction kinetics, is therefore needed for physically complete models of the evolving Earth.

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Methods

Synchrotron methods

Time-resolved *in situ* synchrotron measurements tracked gypsum dehydration under controlled mechanical conditions in the X-ray-transparent Mjöltnir deformation apparatus. X-ray microtomography was performed at PSICHÉ (Synchrotron SOLEIL), I13-2 (Diamond Light Source) and TOMCAT (Swiss Light Source), with isotropic voxel sizes of 1.28, 1.63 and 2.75 μm , respectively. Acquisition intervals varied between facilities, with tomograms collected every 60 s at TOMCAT. Additional loading–unloading experiments at I13-2 used tomography to measure changes in bulk sample geometry, demonstrating that deformation of the unreacted sample was recoverable upon unloading. Reconstructed volumes were segmented into gypsum, bassanite and pores using a common deep learning workflow [1], with reaction stoichiometry providing an independent check on the segmentation. This enabled measurement of reaction progress and evolving microstructure.

Complementary time-resolved X-ray diffraction measurements were acquired at I15 (Diamond Light Source) using a 72 keV monochromatic beam while rotating the sample. Multiple well-resolved reflections from each phase were fitted using the *Continuous Peak Fit* (CPF) software package [2–4]. Integrated peak areas were normalised to initial gypsum and final bassanite values and averaged across reflections to track reactant consumption and product formation. These measurements provide relative indicators of reaction progress rather than absolute phase abundances.

Details of the experiments, the acquisition parameters, diffraction fitting and quality control are provided in the Supplementary Methods.

Uncertainty estimation of kinetic fit

Uncertainty in characteristic reaction times was estimated using 2,000 residual-bootstrap iterations. Residuals from each best-fit Avrami curve were sampled with replacement

and added to the fitted reaction fractions at the original measurement times. Simulated fractions were bounded between 0 and 1, and the Avrami parameters were refitted. The 2.5th and 97.5th percentiles defined the 95% confidence intervals. For the tomography and XRD comparisons, the corresponding hydrostatic best-fit characteristic time was held fixed; these intervals therefore exclude uncertainty in the reference fit. Uncertainties for the gabbroic and peridotite were attained in the same way, but for the mantle sample uncertainties were propagated and the errors for both can be seen as similar.

Change in reaction time

The percentage reduction in characteristic reaction time was calculated as

$$R_{\tau} = 100 * \frac{\tau_{\text{ref}} - \tau_{\text{exp}}}{\tau_{\text{ref}}}, \quad (4)$$

where τ_{ref} and τ_{exp} are the reference and stressed-experiment characteristic times, respectively. Positive values indicate shorter reaction times.

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Author contributions

J.G., D.F. and I.B.B. developed the study concept. J.G., D.F., I.B.B., S.A.H., F.F. and D. Daisenberger designed the experiments. J.G., D.F., E.V.P., R.E.R., I.B.B., D. Duggins, A.K., C.M.S., L.T., D. Daisenberger and F.F. performed the experiments. J.G., D.F.,

450 E.V.P., I.B.B., D. Duggins and S.H. analysed the data. J.G., D.F., I.B.B., D. Duggins
451 and S.H. interpreted the data, while R.E.R., N.B. and F.F. contributed to discussion of
452 the results. J.G. prepared the figures. J.G. and D.F. wrote the manuscript. J.G. and D.F.
453 coordinated and supervised the study. All authors revised the manuscript.

454 **Competing interest declaration**

455 No competing interests.

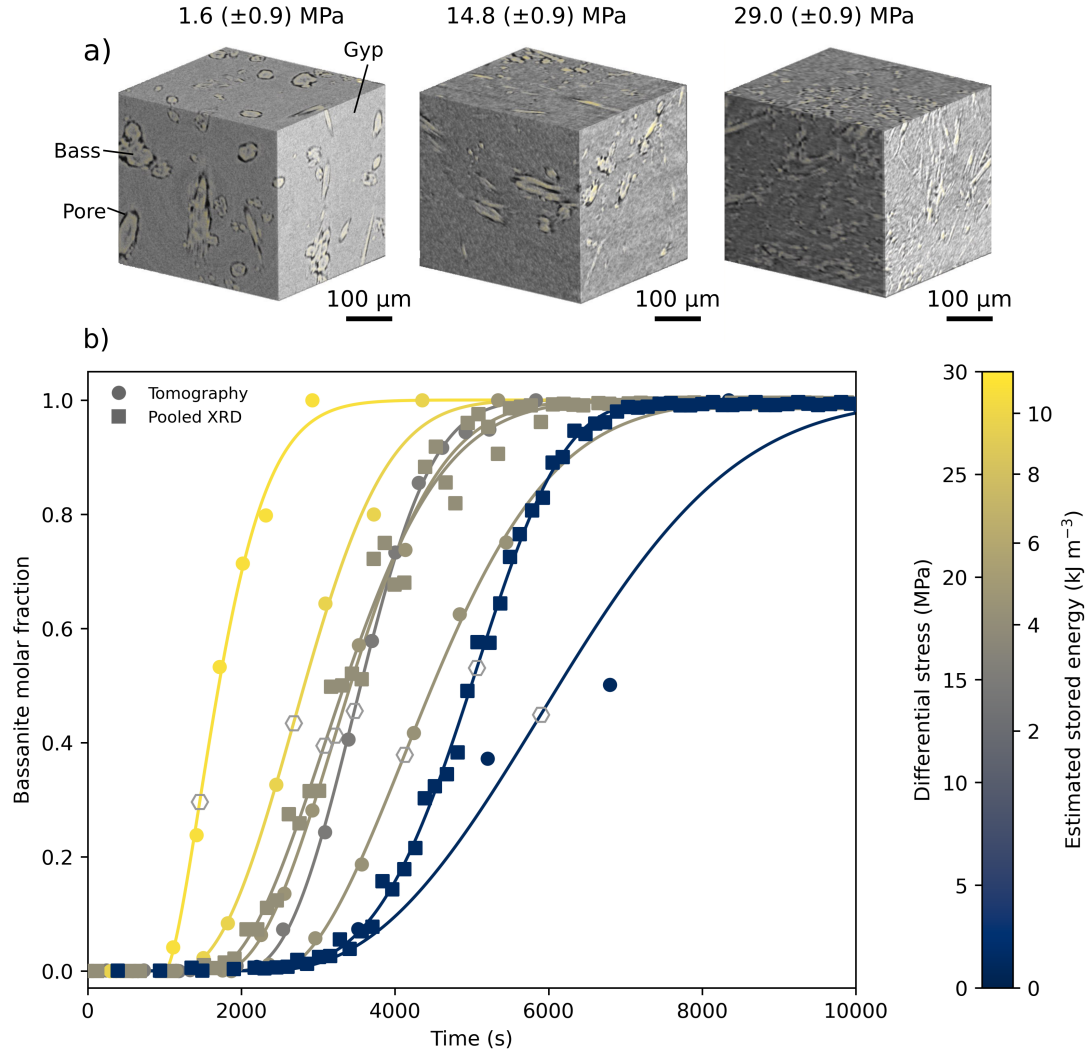


Figure 1: Differential stress impacts gypsum dehydration. a) Representative subvolumes of reaction microstructures at comparable reaction extents ($X \approx 0.2$), showing gypsum (Gyp), bassanite (Bass) and reaction-induced pores. b) Bassanite molar fraction as a function of time for experiments conducted at different differential stresses; solid lines show Avrami fits.

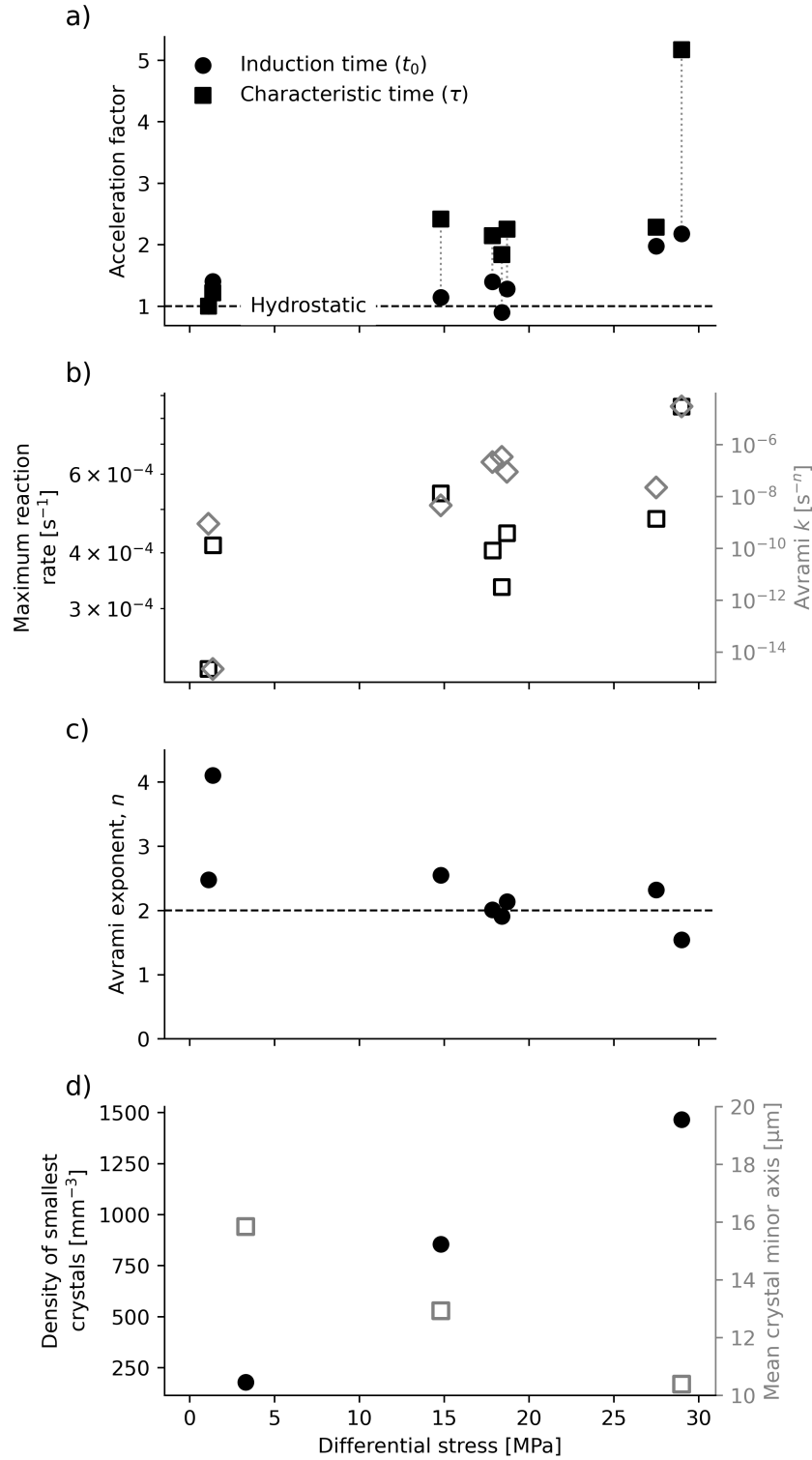


Figure 2: How differential stress changes reaction kinetics. a) Avrami exponent (n); the dashed line = average n . b) Maximum reaction rate (black squares) and Avrami rate constant k (grey diamonds) as a function of differential stress. c) Acceleration factor for the induction time (t_0 ; circles) and characteristic time (τ ; squares), defined as the ratio to the hydrostatic. d) Density of the smallest bassanite crystals and the mean width of the sample of crystals that could be successfully separated from the larger network.

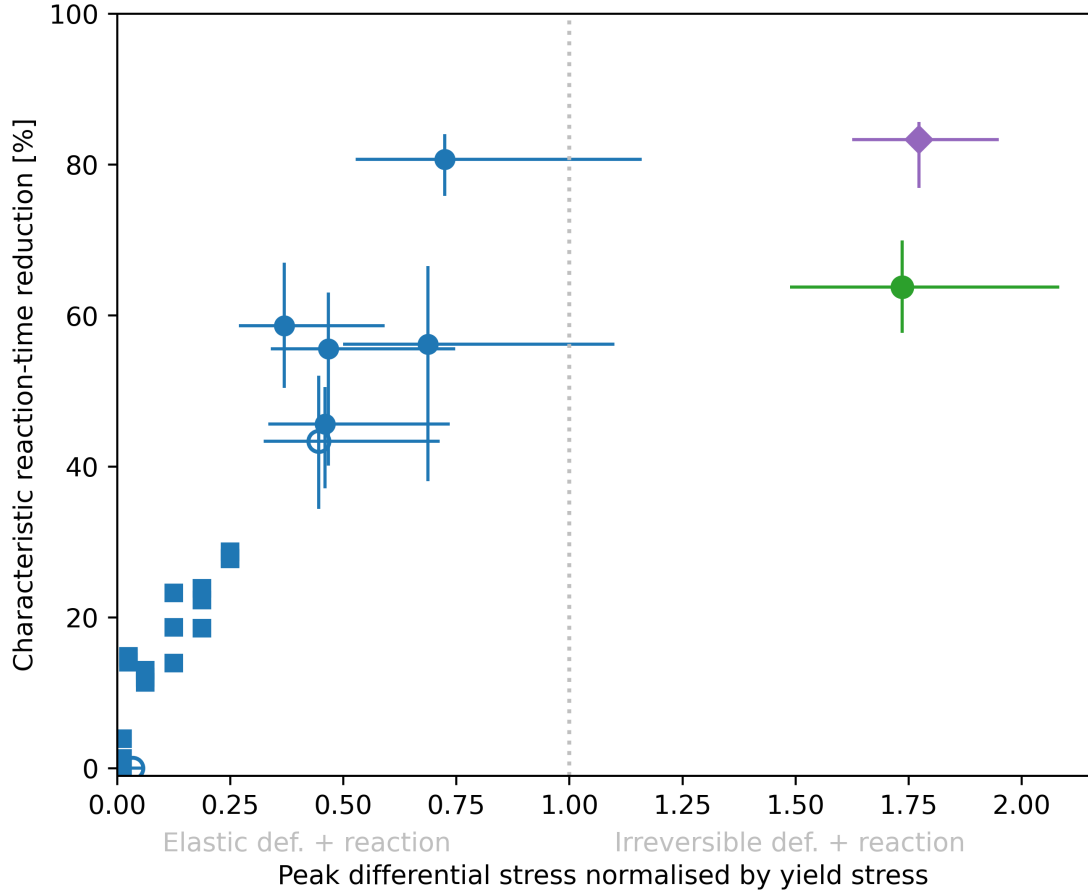


Figure 3: Deformation accelerates reaction kinetics across lithospheric materials. Characteristic reaction-time reduction relative to hydrostatic conditions as a function of peak differential stress normalised by yield stress. Blue symbols = gypsum dehydration (circles(open = XRD) = this study, squares = Schrank et al. [38]); green symbol = garnet peridotite from de Ronde et al. [13]; purple symbol = gabbro from Mansard et al. [39]. Error bars show 95% confidence intervals determined by bootstrap resampling of the Avrami fits and sensitivity brackets for the yield values.

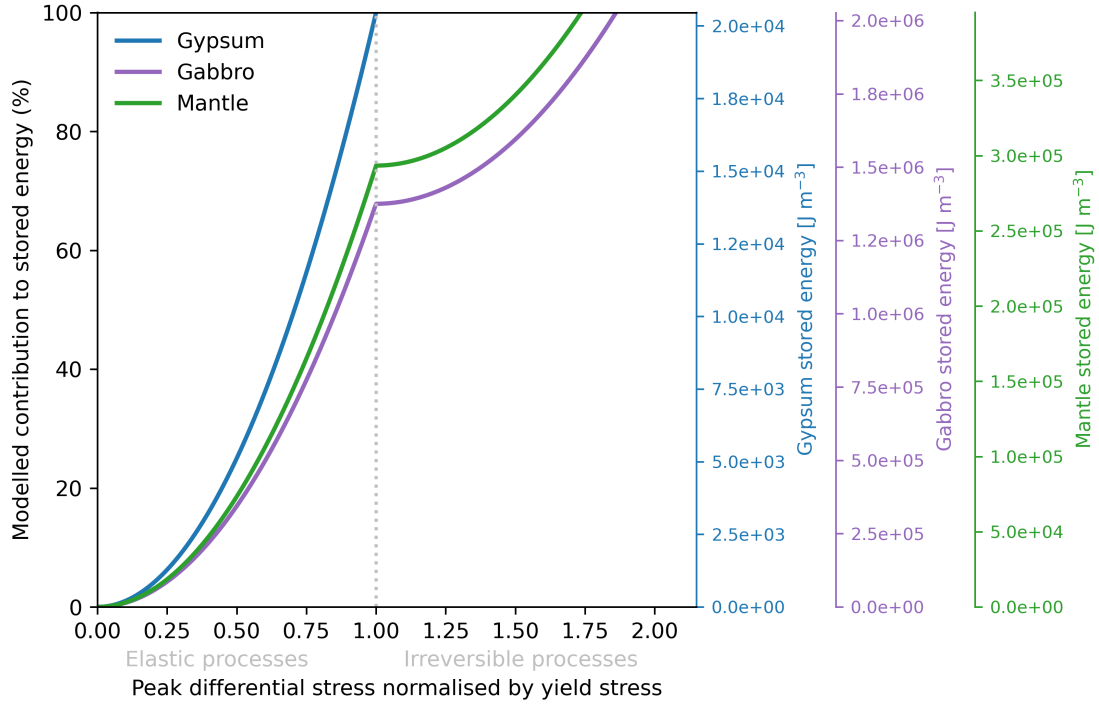


Figure 4: Modelled contribution of deformation to stored energy. Stored-energy evolution as a function of peak differential stress normalised by yield stress for gypsum, gabbro and mantle rocks. Stored energy is calculated from the elastic strain energy below yield and energy retained around dislocations during post-yield deformation. Curves are normalised to their respective maximum elastic stored energies at the yield stress; corresponding absolute stored energies are shown on the coloured axes.

Supplementary Information

The critical role of stored energy in metamorphism

Contents

1	Methods	2
1.1	Experimental samples and conditions	2
1.2	Loading–unloading experiment on unreacted gypsum	2
1.3	Thermodynamic driving force from temperature overstep	2
1.4	Comparison of mechanical relaxation and chemical diffusion timescales	3
1.5	Synchrotron data acquisition	4
1.5.1	TOMCAT — Swiss Light Source	4
1.5.2	PSICHÉ — Synchrotron SOLEIL	4
1.5.3	I13-2 — Diamond Light Source	4
1.5.4	I15 — Diamond Light Source	5
1.6	Synchrotron data processing	5
1.6.1	Tomographic segmentation	5
1.6.2	X-ray diffraction processing	6
1.7	Material properties for the stored-energy model	6

1 Methods

1.1 Experimental samples and conditions

Table S1: Samples and conditions. Temperatures are in °C; pressures and stresses are in MPa.

Sample	Beamline	Method	T_{avg}^1	T_{max}^2	P_c^3 ± 0.2	P_f^4 ± 0.2	σ_{ax}^5 ± 0.8	σ_{diff}^6 ± 0.9
VA10	TOMCAT	μCT	125.3	125.8	20.05	5.04	34.8	14.8
VA_s_1	TOMCAT	μCT	125.0	125.8	20.08	1.06	38.4	18.4
VA_A	TOMCAT	μCT	124.7	125.3	20.08	1.04	38.6	18.7
VA20	TOMCAT	μCT	125.6	126.8	20.08	1.06	47.5	27.5
VA18	TOMCAT	μCT	126.9	127.6	20.08	5.06	49.0	29.0
VA_DC_03	I13-2	μCT	123.4	124.1	20.08	5.06	21.1	1.1
VA_DC_08	I13-2	μCT	58.08	58.60	20.08	5.04	36.30	16.28
DRD_VA1*	PSICHÉ	μCT	119.2	125.0	20.08	5.12	21.6	1.6
LM_XRD_01_VA_H	I15	HEDM	124.17	125.00	20.08	5.08	21.37	1.36
LM_XRD_00_VA_AS	I15	HEDM	124.28	124.80	20.08	5.08	37.79	17.84

¹ Mean recorded temperature over the selected experiment window.

² Maximum recorded temperature over the same window.

³ Maximum confining pressure.

⁴ Maximum pore-fluid pressure.

⁵ Maximum axial stress, calculated using the initial sample cross-sectional area.

⁶ Maximum differential stress ($\sigma_{\text{ax}} - P_c$), calculated at each time point before taking the maximum.

* During experiment heating was set lower to compensate for the beam periodically heating the sample.

1.2 Loading–unloading experiment on unreacted gypsum

Additional loading–unloading experiments at I13-2 used tomography to measure changes in bulk sample geometry, demonstrating that deformation of the unreacted sample was recoverable upon unloading. Figure S1 shows the stress path and the change of the sample length.

1.3 Thermodynamic driving force from temperature overstep

For a small temperature overstep at approximately constant pressure, the Gibbs free-energy driving force can be approximated as $\Delta G \approx -\Delta S \Delta T$. For the experimental thermal overstep, $\Delta T = 125 - 100.6 = 24.4$ K, and taking $\Delta S_{\text{rxn}} \approx 52 \text{ J mol}^{-1} \text{ K}^{-1}$ following McConnell et al. [2],

$$|\Delta G| \approx \Delta S_{\text{rxn}} \Delta T = 52 \times 24.4 \approx 1.27 \times 10^3 \text{ J mol}^{-1}.$$

Using $\rho_{\text{gyp}} = 2310 \text{ kg m}^{-3}$ and $M_{\text{gyp}} = 0.17217 \text{ kg mol}^{-1}$, this corresponds to a volumetric driving force of

$$|\Delta G|_{\text{vol}} = |\Delta G| \frac{\rho_{\text{gyp}}}{M_{\text{gyp}}} = 1.27 \times 10^3 \left(\frac{2310}{0.17217} \right) \approx 1.70 \times 10^7 \text{ J m}^{-3}.$$

Taking the hydrostatic reference differential stress as zero, the maximum increase in stored elastic energy density is calculated using the maximum applied differential stress of 29.0 MPa and Young’s modulus of 39.1 GPa at 125 °C [3]:

$$u_{\text{stored,max}} = \frac{\sigma_{\text{diff,max}}^2}{2E} = \frac{(29.0 \times 10^6)^2}{2(39.1 \times 10^9)} \approx 1.08 \times 10^4 \text{ J m}^{-3}.$$

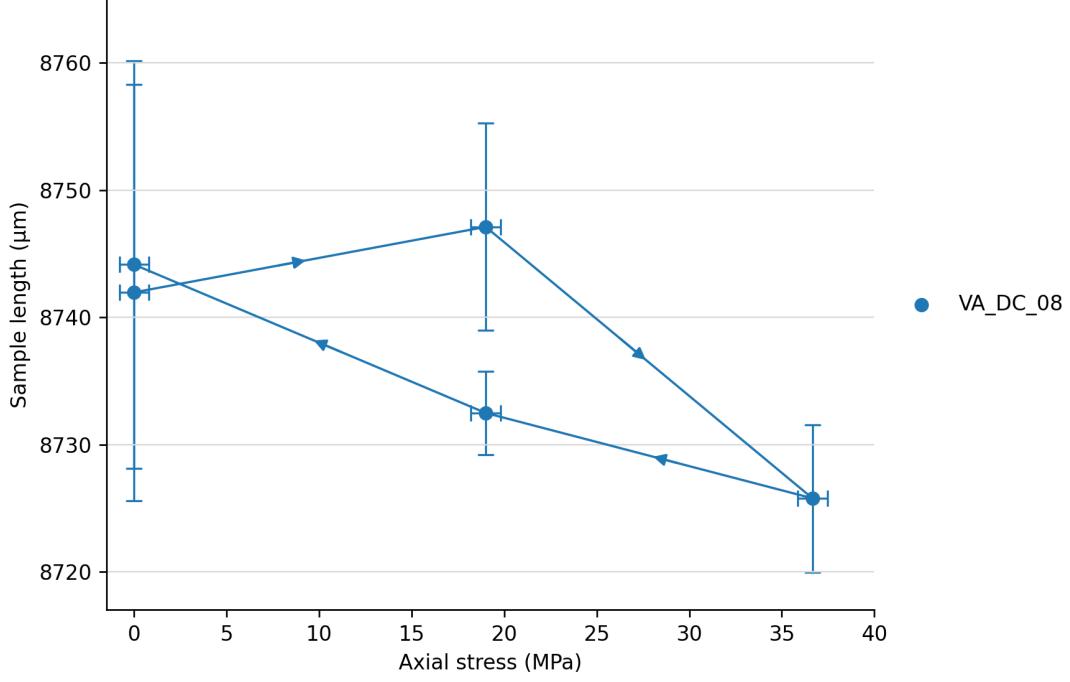


Figure S1: Loading-unloading behaviour of unreacted gypsum measured using X-ray microtomography at I13-2. Uncertainties in sample length are ± 1 standard deviation from measurements made on the left, centre and right of the sample. Stress uncertainty follows the supplementary material of Gilgannon et al. [1].

This is therefore

$$\frac{u_{\text{stored,max}}}{|\Delta G|_{\text{vol}}} \times 100 = \frac{1.08 \times 10^4}{1.70 \times 10^7} \times 100 \approx 0.064\%$$

of the thermodynamic driving force associated with the 24.4 K thermal overstep, or approximately 0.06%.

1.4 Comparison of mechanical relaxation and chemical diffusion timescales

For a Maxwell viscoelastic material, mechanical relaxation is characterised by the Maxwell time, τ_M , while diffusion over a length scale L is characterised by the timescale τ_D . These timescales and their ratio are

$$\tau_M = \frac{\eta}{\mu}, \quad \tau_D \sim \frac{L^2}{D}, \quad \frac{\tau_D}{\tau_M} = \frac{\mu L^2}{D\eta},$$

where η is viscosity, μ is the shear modulus, and D is the chemical diffusivity. Where $\tau_D/\tau_M < 1$, chemical transformation can respond to spatial variations in stored mechanical energy before they relax, whereas for $\tau_D/\tau_M > 1$, mechanical relaxation is faster. Equating the two timescales defines the crossover length

$$L_c \sim \sqrt{\frac{D\eta}{\mu}}.$$

For $L < L_c$, diffusion can operate before mechanical relaxation, whereas for $L > L_c$, mechanical

relaxation is faster. Because $\tau_D \propto L^2$, rocks that behave viscously at macroscopic scales may nevertheless retain stored elastic energy sufficiently long for chemical transformations at grain and sub-grain scales to respond to it.

1.5 Synchrotron data acquisition

Data were acquired during multiple experimental campaigns at different synchrotron facilities and beamlines. Imaging experiments employed filtered polychromatic beam configurations adapted to the characteristics of each beamline, including source properties, optical layout, detector configuration, and experimental geometry. Detailed acquisition parameters for each facility are provided below.

1.5.1 TOMCAT — Swiss Light Source

Four-dimensional (4D) X-ray microtomography datasets were acquired at the TOMCAT beamline using a pink beam with a peak energy of approximately 27 keV. For each tomographic acquisition, 1,500 radiographs were collected over a 180° rotation within 2-4 s. The reconstructed datasets had an isotropic voxel size of 2.753 μm and dimensions of approximately 2016^3 voxels.

Tomographic datasets were acquired every 60 s throughout experiments lasting between 150 and 314 min. Raw radiographs were reconstructed using the TOMCAT reconstruction pipeline, employing propagation-based phase-contrast reconstruction and the Gridrec algorithm.

1.5.2 PSICHÉ — Synchrotron SOLEIL

Experiments at PSICHE were performed using a filtered white beam with a 300 μm tungsten filter, resulting in a peak energy of approximately 65 keV. Imaging was conducted using a 100 μm LuAG:Ce scintillator coupled to a $5\times$ objective lens. Additional attenuation filters were introduced to reduce detector darkening caused by the high X-ray flux.

A sample-to-detector distance of 200 mm was selected as the optimal propagation distance. Longer propagation distances resulted in increased image blurring associated with beam divergence. High-resolution scans were acquired using a Hamamatsu ORCA Flash 4.0 detector, providing an effective voxel size of 1.28 μm . Each tomographic dataset consisted of 4,100 projections collected over a 360° rotation with an exposure time of 15 ms per projection.

The incident beam size was approximately 5.8 mm $\times$ 2.9 mm. Individual reconstructed volumes measured approximately 10.9 mm $\times$ 10.9 mm $\times$ 2.9 mm. Imaging of the full sample required four vertically overlapping scans, with complete acquisition requiring approximately 12 min. Reconstruction and volume stitching were performed using local beamline processing pipelines.

1.5.3 I13-2 — Diamond Light Source

4D X-ray microtomography datasets were acquired at the Diamond-Manchester Imaging Branchline (I13-2) at Diamond Light Source (UK). Imaging was performed in filtered polychromatic beam mode using 1.36 mm pyrolytic graphite and 3.2 mm aluminium filters, producing a peak beam energy of approximately 27 keV.

Images were recorded using a PCO.edge 5.5 detector coupled to a $2\times$ objective (effective magnification $4\times$), yielding a voxel size of $1.625\text{ }\mu\text{m}$ and a field of view of approximately $4.2\text{ mm} \times 3.5\text{ mm}$.

For quasi-static imaging before and after reaction, and during loading and unloading steps, scans consisted of 1,800 projections with an exposure time of 0.2 s per projection, resulting in acquisition times of approximately 6 min per scan. For time-resolved imaging during metamorphic reactions, acquisition speed was prioritised. In these cases, exposure times were reduced to 0.025 s while maintaining 1,800 projections, producing datasets with a total acquisition time of approximately 45 s.

During reaction experiments, data acquisition was restricted to a fixed-height region of interest to maximise temporal resolution. For loading and unloading experiments, the entire sample volume (approximately $3.2\text{ mm diameter} \times 9\text{ mm height}$) was imaged using three to four vertically overlapping scans. Acquisition of a complete sample volume required approximately 18 min. Reconstruction and stitching were performed using local processing pipelines.

1.5.4 I15 — Diamond Light Source

In addition to imaging experiments, time-resolved in situ X-ray diffraction measurements were performed at beamline I15 of Diamond Light Source.

The Diamond storage ring operated at an electron beam energy of 3 GeV and beam currents of approximately 250-300 mA during the experimental campaigns. The beamline was configured in high-energy mode, employing a monochromatic X-ray beam of 72 keV generated by a 3.5 T superconducting multipole wiggler and monochromatised using a Si(111) double-crystal monochromator. The high photon energy ensured sufficient penetration through the pressure vessels and sample assemblies used during the experiments.

At the sample position, the beam was focused to approximately $120\text{ }\mu\text{m}$ (horizontal) $\times$ $80\text{ }\mu\text{m}$ (vertical) using Kirkpatrick-Baez mirrors. Following exit from the vacuum beam pipe through a diamond window, clean-up slits and a tungsten collimator located upstream of the sample were used to minimise air scatter and parasitic background. A 3 mm diameter tungsten beamstop was positioned downstream of the sample to shield the detector from the transmitted beam.

Diffraction data were acquired during continuous rotational sweeps over 90° at multiple sample positions throughout the dehydration experiments.

1.6 Synchrotron data processing

1.6.1 Tomographic segmentation

Tomographic datasets were segmented following the workflow described by Rizzo et al. [4], which builds upon the image processing procedures detailed by Gilgannon et al. [1]. The same processing framework was applied across datasets acquired at the different synchrotron facilities.

1.6.2 X-ray diffraction processing

Time-resolved X-ray diffraction data were acquired throughout the dehydration experiments using continuous acquisitions, while rocking the cell around the vertical axis to increase count statistics. Prior to peak fitting, detector geometry was calibrated in Dioptas [5] using a LaB_6 reference standard; the wavelength of monochromatic X-rays was 0.172 Å. Data preprocessing was limited to detector-geometry calibration and masking of the beamstop, detector-module gaps and dead pixels.

Diffraction peaks were fit using the *Continuous Peak Fit* (CPF; <https://github.com/ExperimentalMineralPhysics/Continuous-Peak-Fit>) software package (as used by Chen et al. [6], Daniel et al. [7] and Zhang et al. [8]). The package fits continuous models for diffraction peaks directly to the as-collected diffraction data without resampling the detector pixels. The data is fit with a pseudo-Voigt peak the parameters of which are formulated as Fourier series in azimuth, the orders (number of parameters) of which are tuned to the data.

Six or seven intense and well-resolved reflections were selected for both gypsum and bassanite and tracked throughout the experiments. Gypsum reflections were fitted in chronological order, following the progressive disappearance of the reactant phase. Bassanite reflections were fitted in reverse chronological order, as CPF was found to track progressively decreasing peak intensities more robustly than weak peaks emerging from background noise.

All fitted datasets underwent automated and manual quality-control procedures, including inspection of fitted peak positions, peak profiles and residuals. Datasets affected by poor convergence, severe peak overlap or insufficient signal-to-noise ratios were excluded where appropriate.

For each fitted reflection, peak intensity and integrated peak area around all azimuths were extracted as a function of time and normalised relative to the first acquisition after crossing the phase boundary (100.6 °C) for gypsum and the last acquisition before cooling down the experiment for bassanite. The temporal evolution of individual reflections was then assessed from the resulting normalised intensities and peak areas.

Phase evolution was estimated by combining the results from multiple reflections belonging to the same phase. Averaging across several independent reflections reduced the influence of fitting uncertainty and local peak-overlap artefacts, yielding a more robust measure of the relative evolution of gypsum and bassanite during dehydration. These phase-evolution curves should therefore be interpreted as relative indicators of reaction progress rather than as quantitative phase abundances derived from full quantitative phase analysis.

1.7 Material properties for the stored-energy model

Table S2: Values used in stress normalisation. Yield thresholds are adopted from published mechanical data and visual interpretation of loading curves. The stated brackets illustrate sensitivity to the adopted thresholds and are not statistical uncertainties.

Material	Yield stress (MPa)	Peak stress (MPa)	Source
Gypsum	≈ 40 (± 15) ^a	29.0 ^b	This study; Brantut et al. [9]
Gabbro	≈ 550 (± 50)	975.2 ^c	Mansard et al. [10]
Peridotite	≈ 300 (± 50)	521 ^d	de Ronde & Stünitz [11]

^a A gypsum yield threshold of 40 MPa was adopted, informed by the peak/yield differential stresses reported by Brantut et al. [9]: approximately 37–42 MPa at 10 MPa confining pressure and 46–51 MPa at 20 MPa confining pressure, at room temperature or 70 °C. These measurements do not directly determine yield under our experimental conditions. A sensitivity bracket of 25–55 MPa was therefore adopted. Fracturing observed near 30 MPa in our experiments provides additional context but does not independently establish the elastic yield threshold.

^b Maximum differential stress reached in experiment VA18.

^c Mean peak differential stress of the five experiments represented in the deformed kinetic dataset: OR47NM (989 MPa), OR51NM (901 MPa), OR49NM (1111 MPa), OR41NM (1094 MPa), and OR34NM (781 MPa), as reported in Table 2 of Mansard et al. [10]. The adopted yield threshold was estimated visually from the loading curves in Fig. 12a. The 500–600 MPa bracket illustrates sensitivity to this choice.

^d Peak differential stress reported for experiments 32AA and 28AA in Table 1 of de Ronde and Stünitz [11]. The adopted yield threshold was estimated visually from the loading curves in Fig. 4b. The 250–350 MPa bracket illustrates sensitivity to this choice.

Table S3: Elastic properties used in the stored-energy model.

Material	E (GPa)	μ (GPa)	Source
Gypsum	39.1 ^a	14.7 ^a	Brantut et al. [3]
Gabbro	110 ^b	43 ^b	Christensen & Salisbury [12]
Peridotite	153.4 ^c	60 ^c	Christensen [13]

^a Brantut et al. [3] report reference bulk and shear moduli for gypsum of $K_0 = 41$ GPa and $\mu_0 = 16$ GPa. These were corrected to the experimental temperature of 125 °C using the mean measured temperature dependencies $\partial K/\partial T = -36.1$ MPa °C⁻¹ and $\partial \mu/\partial T = -12.7$ MPa °C⁻¹, giving $K_{125} = 37.4$ GPa and $\mu_{125} = 14.7$ GPa. Young’s modulus was calculated as $E = 9K\mu/(3K + \mu)$, giving $E_{125} = 39.1$ GPa.

^b Gabbro elastic moduli were derived from the shear-wave velocity $V_S = 3.8$ km s⁻¹ and Poisson’s ratio $\nu = 0.30$ reported by Christensen & Salisbury [12]. Assuming $\rho = 3.0$ g cm⁻³, $\mu = \rho V_S^2 = 43.3$ GPa and $E = 2\mu(1 + \nu) = 112.6$ GPa. Values of $\mu = 43$ GPa and $E = 110$ GPa were used in the model.

^c Christensen [13] reports elastic properties for unaltered peridotites at 2 kbar of approximately $\mu = 57$ –62 GPa and $E = 147$ –159 GPa. Representative values of $\mu = 60$ GPa and $E = 153.4$ GPa were used in the model.

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