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Mineral host inclusion systems are a window into the solid-state rheology of the Earth

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Natural processes such as earthquakes, volcanism and orogenesis are controlled by plate tectonics which, in turn, depend on how rocks and minerals, the building blocks of Solid Earth, deform under different environmental conditions. The rheology of rock-forming minerals is therefore the key for understanding the geodynamics of our planet. Our present knowledge of mineral rheology mainly comes from laboratory experiments and theoretical models that are based on synthetic systems with simplified chemistry. However mineral properties strongly depend on structural defects and impurities. Therefore, the interpretation of natural chemically complex systems requires uncertain extrapolations. Mineral inclusions are macroscopic defects with respect to their host, so studying in situ their effects on the rheology of their host minerals opens a promising prospect for a better understanding of the rheology of mineral assemblages and thus the dynamics of our planet.

The interpretation of large-scale geodynamic processes, such as plate tectonics, relies on our understanding of mineral rheology at pressure (P) and temperature (T) (e.g. ref. 1). Deformation experiments performed on minerals and rocks under various thermodynamic conditions in the laboratory are the main approach used to understand mineral rheology. However, most experimental results rely on ex-situ characterization carried out on samples quenched from high P - T conditions to ambient conditions. Consequently, direct information on actual deformation mechanisms is lost. Furthermore, studies on polycrystalline materials carry intrinsic uncertainties about stress distributions within the sample, due to grain boundaries, grain interactions, mineral anisotropy, and heterogeneous loading conditions during the experiment (e.g. ref. 2). Thus, reconstructing the magnitude of stress that triggered the deformation is impossible even for simple and ideal systems, let alone that of a whole rock comprised of many phases.

Mineral inclusions, why do they matter?

Crystal defects such as dislocations, chemical impurities and vacancies play a crucial role in influencing the elasticity and properties of minerals and their rheology. These defects act as perturbations of the crystal structure and as powerful catalysts of non-elastic processes because they reduce the activation energy of deformation and solid-state flow by several orders of magnitudes (e.g. refs. 1,3). In addition to defects and vacancies, minerals often contain fluid, melt and solid (crystalline or amorphous) inclusions that can reach significant volumetric abundances and strongly affect the elastic

properties (and thus the mechanical properties and rheology) of the host crystal. While the investigation of natural vs. synthetic crystals requires caution in the interpretation of the results, the investigation of mineral host crystals containing inclusions provides a new benchmark for a more comprehensive and improved understanding of mineral and rock rheology.

Beyond the complexity introduced for these systems at the micro-to-nano scale, host-inclusion mineral systems represent the mechanically simplest “rock sample” occurring in nature in which the effects of grain boundaries between crystals and the contrast in elastic properties can be explored. Moreover, host-inclusion minerals represent apparatus-free “pre-stressed” systems due to the contrast between their thermal expansion and compressibility coefficients that, after entrapment, lead to residual pressures (P_{inc}) in the inclusions that differ from the external pressure (e.g., refs. 4,5). By changing the external pressure and temperature applied to the host crystal, the residual pressure in the inclusion is also changed, and this allows us to explore the effect of different stress conditions on the rheology of the minerals at various P and T . Fundamental to this approach is our ability to quantify in situ the residual pressure of a mineral inclusion at non-ambient conditions by Raman spectroscopy.

Using Raman spectroscopy to estimate residual pressure P_{inc} in mineral inclusions

Raman spectroscopy is a technique based on the inelastic scattering of light by the collective atomic vibrations in crystals with characteristic (quantized) energies (i.e. phonons) (e.g. ref. 6). The frequencies ω of the atomic

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vibrations in crystals depend on the interatomic interactions and thus carry structural information about the minerals. A Raman spectrum consists of a characteristic pattern of “Raman peaks” whose positions (usually given in cm^{-1}) represent the frequencies of specific atomic vibrations (phonon modes). The Raman spectrum is thus a ‘finger-print’ of a mineral. Under high pressure, atoms get closer together because the volume is reduced so the forces between the atoms change, leading to different vibrational frequencies from those of a free crystal at ambient conditions. Therefore, the difference between the Raman peak positions of a mineral inclusion with respect to those of a free crystal taken as reference ($\Delta\omega = \omega_{\text{inc}} - \omega_{\text{ref}}$) is proportional to the accumulated elastic strain in the inclusion, from which the residual pressure P_{inc} can be derived. There are two ways to calculate the P_{inc} starting from measured $\Delta\omega$: the hydrostatic approach and the Grüneisen-tensor approach. In the hydrostatic approach, the P_{inc} is directly calculated from the measured $\Delta\omega$ using the Raman shifts of the same mineral measured, usually in diamond-anvil cells, at different hydrostatic pressures (e.g. ref. 7). This approach assumes that deviatoric stresses in the inclusion caused by its mechanical interaction with the crystalline host are negligible. On the other hand, the Grüneisen-tensor approach considers the intrinsic anisotropy of the inclusion. In this case, one obtains first the residual strain of the inclusion and then the P_{inc} as the negative of the mean normal stress, calculated from the strain via the elastic properties of the inclusion^{8,9}. The P_{inc} of the inclusion and its evolution at non-ambient conditions measured in this way can be used to constrain the rheology of minerals.

Host-inclusion systems as a benchmark for investigating mineral and rock rheology

Fluid, melt, and solid inclusions within minerals provide valuable insights into the geological history and formation conditions of rocks (e.g. ref. 10). Such inclusions, encapsulated within host minerals during their growth, offer a unique record of the physical and chemical environments prevailing at the time of mineral formation. The volumetric abundance of inclusions within a mineral host may range from <0.1% up to 50%, and their potential for affecting the elastic properties of the mineral host is currently under investigation. An increasing number of experiments and numerical models focused on the investigation of mineral inclusions in mineral hosts show

how the rheology of the host can dramatically change depending on the elastic properties and the stress state of the encapsulated inclusions (e.g. ^{11–13}).

Zircon and quartz inclusions in garnet are common occurrences in metamorphic rocks. Zircon (ZrSiO_4) is an accessory and chemically inert mineral and is usually not involved in metamorphic reactions with other rock-forming minerals. On the other hand, quartz (SiO_2) is a major rock-forming mineral, easily dissolved in metamorphic fluids and involved in many chemical reactions. Garnet [$(\text{Fe}^{2+}, \text{Mg}, \text{Ca}, \text{Mn})_3(\text{Al}, \text{Fe}^{3+})_2\text{Si}_3\text{O}_{12}$] is a typical high-pressure phase that crystallizes from the breakdown of other minerals that are stable at lower pressures. During crystallization, garnet grows, forming porphyroblasts and trapping pre-existing minerals such as zircon or phases that are involved in the garnet-forming reaction, such as quartz.

Zircon inclusions in garnet (ZiG) always show a positive residual pressure at ambient conditions, whereas quartz inclusions in garnet (QuiG) can show either positive or negative residual pressure. Such a difference arises from the different contrast in the thermal expansion and compressibility coefficients between zircon and garnet and between quartz and garnet. This is the key to *unlock* the rheology of minerals under non-ambient conditions.

For example, because the thermal expansion of zircon is lower than that of garnet, heating ZiG systems leads to residual volume strain in the inclusion that changes from negative to positive values, corresponding to positive and negative inclusion residual pressures (P_{inc}), respectively (Fig. 1). Consequently, the garnet host surrounding zircon inclusions experiences tensile stress conditions at high temperature. The volume strain of the zircon inclusion measured on heating using in situ Raman spectroscopy can be predicted by elastic models up to a given temperature (T^I in Fig. 1) corresponding to a given amount of tensile stress. For further temperature increments, however, the measured strain does not continue to follow the prediction of the elastic model but becomes zero (T^{II} in Fig. 1). This shows that the system experiences visco-plastic relaxation under tensile stress as confirmed by the development of dislocation-like textures in the garnet surrounding the inclusion. Volume strain measured on subsequent cooling follows a completely different path, which always extends within the

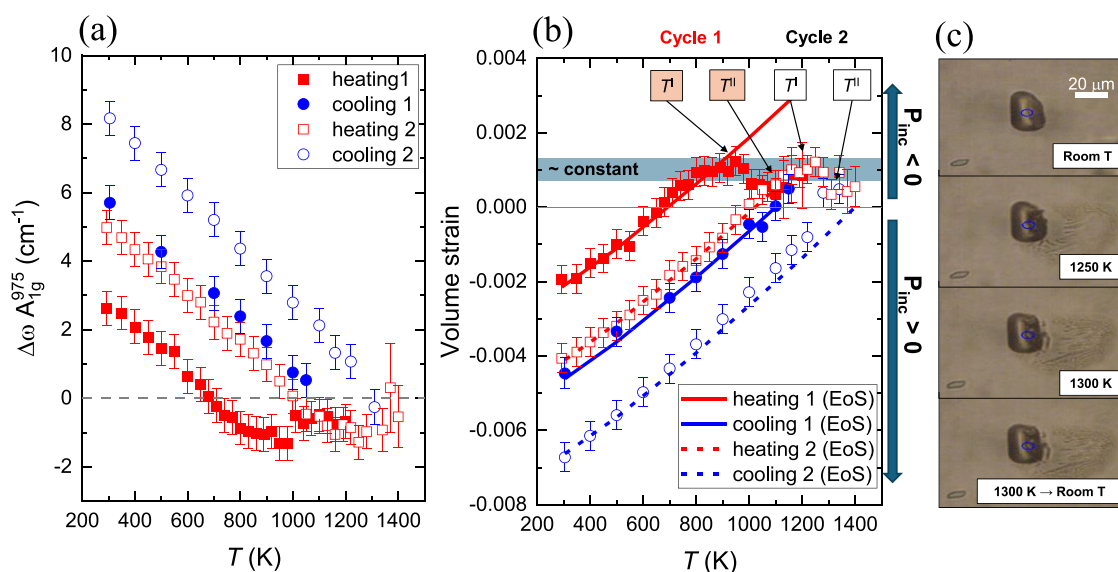


Fig. 1 | Stressed zircon inclusions within garnet (ZiG) under high temperature. **a** Difference in the measured peak position ($\Delta\omega$) of a zircon inclusion in garnet from a free crystal at the same temperature during two consecutive heating-cooling cycles as determined from in situ high-temperature Raman spectroscopy at ambient pressure (average error bar $\pm 0.49 \text{ cm}^{-1}$ and 0.1 K). **b** Volume strain of the inclusion calculated from the measured $\Delta\omega$ (average error bar $\pm 4\text{E}-04$ and 0.1 K). Solid and dashed lines are volume strain predictions from elastic models. Because the peak

position and volume strain are not reversible, these trends represent non-elastic processes occurring on the time scale of laboratory experiments. Raman spectra on the inclusion were collected with a laser power < 14 mW to avoid laser heating of the inclusion during measurements (e.g. ref. 12). **c** Optical micrographs under plane-polarized light of a zircon inclusion within garnet showing dislocation-like textures developed on heating, when garnet was under tensile stress conditions, and preserved on subsequent cooling to room temperature.

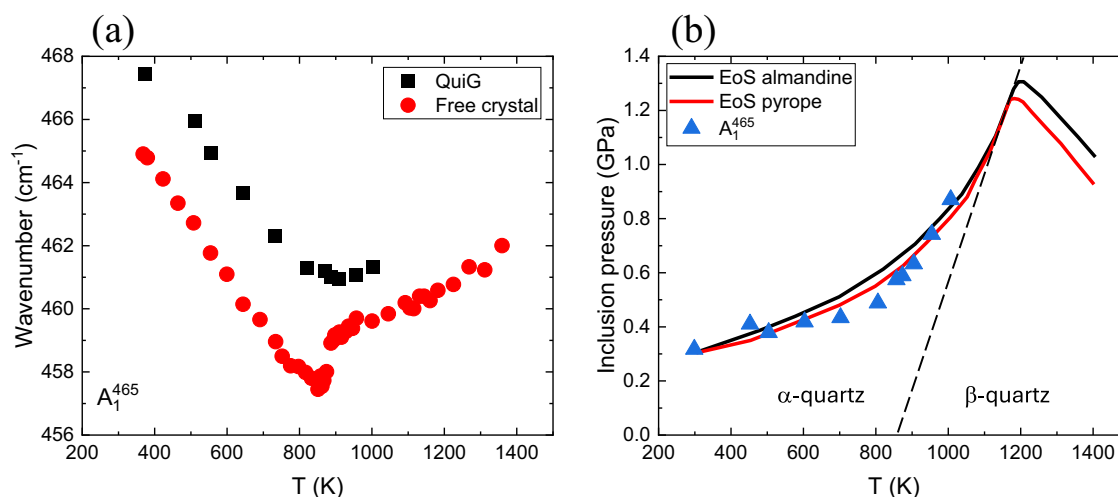


Fig. 2 | Stressed quartz inclusions within garnet (QuiG) under high temperature. **a** Temperature evolution of Raman peak position (wavenumber) for the A_1 mode near 465 cm^{-1} of free (red circles) and entrapped (black squares) quartz crystals (average error bar $\pm 0.35\text{ cm}^{-1}$ and $\pm 0.1\text{ K}$). Above 1000 K the quartz inclusion triggered catastrophic embrittlement of the host garnet when approaching the temperature of the α - β phase transition. **b** Expected pressure (P) evolution of

a quartz inclusion calculated with almandine and pyrope hosts, plotted as lines (average error bar $\pm 0.1\text{ GPa}$ and $\pm 0.1\text{ K}$). Solid symbols are the pressures calculated by applying the hydrostatic calibration to a linear fit of the free quartz data. The dashed line represents the α - β phase transition boundary (redrawn and modified after ref. 14).

compressive stress regime with no relaxation. Rather, by considering the resetting of inclusion P_{inc} at the temperature T^{I} (Fig. 1), the volume strain measured on cooling can be predicted via elasticity again, and at room temperature, the zircon P_{inc} is different from the starting value before heating. Further heating-cooling cycles show that tensile stress on the garnet triggers fast (within a few seconds) plastic deformation, but similar levels of compressive stress generated on cooling do not lead to any deformation. These experimental results demonstrate that, besides temperature, the sign of the applied stress also plays a crucial role in the visco-plastic relaxation of minerals and should be considered when addressing mineral rheology under non-ambient conditions.

On the other hand, because the thermal expansion of quartz is larger than that of garnet, heating QuiG systems leads to an increase in the inclusion residual pressure (Fig. 2). For example¹⁴, have shown by in situ Raman spectroscopy at high temperatures that pressurized quartz inclusions in an almandine-rich garnet may trigger catastrophic embrittlement of the host when the temperature conditions approach those of the α - to β -quartz phase transition. Indeed, during such a transition, the thermal expansion of the inclusion is expected to notably increase (e.g. ref. 15), resulting in a large amount of stress accumulating at the host-inclusion boundary that triggers brittle failure of the host¹⁴.

These results demonstrate that different inclusions, with markedly different physical properties, can be used to explore the effect of different stress states (tension vs. compression) on the rheology of the surrounding host mineral at the same external P - T conditions.

On a large scale, tectonic overpressure and underpressure are important phenomena in lithospheric geodynamics and metamorphism, and their magnitude may range between $\pm 100\%$ of the lithostatic pressure and up to GPa levels (e.g. ref. 16). Such magnitudes strongly depend on the contrast of the rheological properties of deforming rocks. A typical geodynamic environment where both overpressure and underpressure domains may occur are subduction zones. Thus, despite the fact elastic and rheological properties of a mineral like garnet are supposedly well-known, our observations on host-inclusion systems show that deviations from *ideal* conditions may result in extreme behavior opposite to what is expected that profoundly impacts our understanding of mineral and rock rheology.

Moreover, the geologic relevance of host-inclusion system rheology makes them even more attractive to the scientific community. The inclusion residual pressure measured at ambient conditions is often applied to constrain the history of metamorphic rocks via the elastic thermobarometry

method (e.g., refs. 7,17,18), but changes in the rheology of minerals affect the results of thermobarometric estimates¹².

Outlook

Solid inclusions, entirely trapped within their host, provide a great opportunity to study the rheology of rock-forming minerals under various environmental conditions. Such different thermodynamic environments can be explored by putting a host-inclusion system within a resistivity-heated diamond anvil cell (RH-DAC) and measuring in situ the evolution of the inclusion residual stress at various P - T conditions. The measurement of inclusion residual strain is currently routine, and it is carried out via Raman spectroscopy and/or single-crystal X-ray diffraction (e.g. ref. 8). Therefore, the rheology of the system can be explored by following two approaches:

1. by monitoring how the inclusion residual pressure changes as a function of different P - T conditions against predictions calculated using the elastic models based on the mineral equations of state (EoS) (e.g. ref. 19), and
2. by monitoring how the inclusion pressure changes as a function of time at fixed P and T conditions to determine the effects of viscous relaxation.

Such experiments performed on natural host-inclusion systems can be used to address several aspects of mineral rheology, including the following.

The effect of stress state

Given that quartz is softer than garnet, whereas zircon is stiffer than garnet, quartz and zircon inclusions in garnet allow one to explore the effects of different stress states of the host mineral at the same external P - T conditions. Therefore, by exploring the behavior of quartz and zircon inclusions in garnet at P - T conditions corresponding to realistic metamorphic gradients, it is possible to determine the garnet rheology relevant to natural metamorphic processes. The stress state of host garnets can be further modified by using polymorphic phase transitions of the inclusions, such as the α - β transition in quartz (Fig. 2). Since such transformations involve changes in the crystal atomic structure, they result in changes in the volume as well as in the elastic and viscous/plastic properties of minerals (e.g., refs. 1,3). A clear and well-known example of this process is the common occurrence of radial cracks surrounding coesite (a high-pressure polymorph of SiO_2) inclusions partially transformed to quartz (the low-pressure

counterpart). The ca. 10% volume increase during the coesite → quartz transformation leads to stress levels around the inclusion that cannot be supported by the host garnet, which, consequently, breaks and allows the inclusion to expand and relax²⁰.

The effect of crystal chemistry

Garnet can accommodate chemical substitutions at all three types of cation sites. Therefore, samples coming from different rocks having different bulk chemistries generally contain garnets with significantly different chemical compositions. For instance, the strength of garnet scales with the shear modulus and melting point²¹. Given that garnets with different compositions have different shear moduli and melting points, the ubiquity of QuiG and ZiG systems from many metamorphic rocks with different bulk compositions allows one to explore the effects of chemistry on the garnet rheology. This is fundamental because garnets with different chemical compositions come from different geodynamic contexts. Therefore, by knowing the mechanism of deformation of this rock-forming mineral we can get a clearer understanding of the geological processes that are involved. Moreover, our method will allow us to define the mechanical properties of mineral solid solutions that so far cannot be described using a unique theory (e.g. refs. 22,23).

The effect of chemical environment

By chemical environment, we refer to the fugacity of key chemical components such as H₂O, O₂, CO₂, etc. all of which are important in geological processes involving fluid migration. The chemical environment has an important effect on the rheology of minerals because chemical components affect the defects in the crystal structure and, in turn, the movement of dislocations, especially at high temperatures. For example, the effect of water fugacity in the rheology of a polycrystalline garnet sample at simultaneous high *T* and *P* can be studied in situ using a synchrotron radiation in a *deformation-diamond* apparatus (D-DIA)²⁴. In a RH-DAC this kind of experiment is difficult, or even impossible, to be performed accurately because the “chemical environment” is mainly controlled by the pressure-transmitting medium and to avoid tarnishing of the anvils at high temperatures, the system needs to be flushed via mildly reducing gas such as Ar. In this case the addition of water or any different chemical agent would be very dangerous. The effect of oxidizing versus reducing chemical environments at elevated temperatures can be addressed using suitable high-temperature stages that can operate at different stages of vacuum and/or allow to purge of the sample chamber (e.g. ref. 25). As commercially available humidity controllers operate at mild temperatures, a different approach to the problem of addressing the effect of “water” should be applied, for example using garnet hosts with different amount of OH groups (i.e. hydro-garnet components).

The effect of geometry and mutual crystallographic orientation of host and inclusions

The inclusion shape is expected to play a role in the deformation processes because of stress concentrations at the corners and edges of the inclusion that lead to heterogeneous stress distribution in the host garnet^{26–28}. In this regard, in situ experiments performed via Raman spectroscopy are particularly suitable because they can provide local information about the strain of the garnet on a micron scale. Moreover, we also expect the mutual crystallographic orientation between the host and the inclusion minerals to play a role because garnet crystals have specific slip systems along which the development of non-elastic deformation is enhanced^{21,28}. In principle, viscoplastic deformation only occurs if a given amount of shear stress is resolved along the slip system. Consequently, the crystallographic orientation of anisotropic crystals (which develop anisotropic stresses) with respect to the slip systems of the host can influence the non-elastic relaxation. XRD measurements of the host-inclusion system before the HP-HT experiments can be used to define the crystallographic relation between the two crystals and give a better understanding of possible heterogeneity in the non-elastic relaxation processes.

Therefore, the systematic exploration of the rheology of garnet, a major mineral in the lower crust of tectonic plates, by using host-inclusion systems will help the scientific community to develop an understanding of the rheological properties of minerals across the *P–T*-stress conditions that are important in many geodynamic settings. Moreover, a new set of single-crystal flow-law equations can be provided for each host mineral investigated and compared with those of the polycrystalline counterpart to better understand the role of grain boundaries in the rheology of geomaterials, a highly debated topic.

Data availability

Data are available at the following link: https://figshare.com/articles/figure/Figure_1_for_repository_opju/27075322?file=49318450.

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Author contributions

N.C. and M.A. conceived the manuscript, N.C. wrote the first draft of the manuscript, N.C., R.J.A., B.M. and M.A. equally contributed to editing the first draft of the manuscript.

Competing interests

The authors declare no competing interests.

Additional information

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