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Uniaxial compression of calcite single crystals at room temperature: insights into twinning activation and development

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Supplementary data

This supplementary data reports some preliminary experiments carried out on Iceland Spath crystals ahead of the experiments described in the article with the aim at determining the suitable loading conditions to be applied to the strain-free synthetic calcite crystals, purchased from SurfaceNet supplier. As explained in the article, the purpose of the study is to constrain the critical resolved shear stress (CRSS) for twinning activation, and therefore it is needed to apply an uniaxial stress state to a completely strain-free crystal. However, Iceland Spath crystals always contain a few cleavages and/or twinned planes, in addition to substantial amounts of micro-fluid inclusions, which precludes the possibility to precisely infer the desired CRSS value. They were consequently used to establish the most suitable deformation protocol in terms of sample preparation, loading rates, and conditions for in the situ optical/SEM monitoring. This preparation phase allowed for instance to select the best appropriate CCD cameras and to establish the SEM imaging conditions. The results presented in this supplementary data are the one with the same experimental conditions than in the main text.

1 Sample 4 x 4 x 8 mm^3 compressed along $[01\bar{1}0]$

Figure 1 shows the loading curve of the first experiment carried out on a small synthetic sample (4 x 4 x 8 mm^3), compressed along $[01\bar{1}0]$ (configuration 1, shown in Fig. 1 in the article main text). The specimen was first deformed until 1% shortening, reaching stage IIIa (plastic deformation with constant hardening). The very first twins were detected at about 0.7% shortening. Twin activity progressively increased during strain hardening, which is indicated by the numerous drops of axial stress (serrations). After reaching 0.85% shortening the specimen was completely unloaded and removed for observations, prior to a second compression test. During the second loading, the sample rapidly reached the stage IIIb, characterized by nearly steady state flow stress. This “plateau-like” curve is serrated, composed of numerous and regular segments, corresponding to strain hardening stages followed by sudden stress drops. The test was stopped at about 15 % shortening (Fig. 1). The sample entirely broke apart after removing from the uniaxial press. During the stage IIIa, the sample was removed from the uniaxial press to determine the twin lamellae crystallographic orientation using EBSD. This orientation was used to calculate the Schmid’s factor and the associated applied resolved shear stress and particularly the value at the onset of twin activation (CRSS). The results show (Fig. 1) that for all the samples the expected twin lamella family was activated during the experiments. For the present orientation, with loading along $[01\bar{1}0]$, the calculated Schmid’s factor is 0.39 and the corresponding CRSS is 0.66 MPa. This value is only slightly larger than the CRSS obtained for the sample with the same lattice orientation with respect to loading direction, but of smaller size (3 x 3 x 6 mm^3) described in the main text.

2 Iceland Spaths

2.1 "Pita" Sample

The sample is a parallelepiped of 3.16 x 5.26 x 5.71 mm^3 in size. The applied constant loading rate is of 5 x 10^{-5} mm/s (along the greatest axis), which results in a uniaxial strain rate of about 10^{-5} s^{-1}. In Figure 2 are represented the results of the experiment. The different stages of deformation are indicated, like for the others samples. An early micro-fracture developed during the stage I (phase-in or emplacement stage) and ran across the entire crystal by the end of the stage I (Figure 2B). The early initiation of this fracture can be explained by a lack of parallelism of the loaded sample faces, leading to stress concentration at the bottom end of the sample. Accordingly, this part of the sample is also the first to show twinning (1.1% strain, Figure 2C). Later on a second quasi-vertical fracture developed and the sample may have been split onwards into distinct parts, as suggested by the distinct evolution of twins on both sides of the fractures. The upper part of the crystal twinned later (Figure 2D) at a higher stress value, before reaching the "plateau-like" part of the curve (Figure 2E). The latter is composed of numerous and regular segments corresponding to strain hardening stages followed by sudden stress drops. This part of the curve corresponds to the highest densification of the twinned planes in the upper part of the crystal, but also to the development of a network of micro-fractures. The small bottom corner part did not show any twin densification, suggesting de-cohesion from the larger upper part, which remained the load bearing part of the crystal. This fracture network finally led to the collapse of the sample at the end of the experiment. The "Pita" sample has been cut from the same Iceland Spath and is supposed to have the same orientation than "Johnny", within relative degrees of Johnny's preparation. Then, the Schmid factor is supposed to be about 0.16. The applied stress at the appearance of the first twin lamella is 20 MPa in the upper part of the crystal still holding the stress. Accordingly, the CRSS value is calculated at about 3.2 MPa.

2.2 "Johnny" Sample

The sample is a parallelepiped of 3.58 x 3.75 x 6.57 mm^3 in size. The loading rate during the uniaxial deformation is of 6 x 10^{-5} mm/s (along the greatest axis, 6.57 mm), which results in a uniaxial strain rate of about 10^{-5} s^{-1}. In this experiment, the phase-in period (stage I) is quite long due to an obvious lack of parallelism of the left hand loaded face. The progressive emplacement until the whole face becomes in contact with the piston is shown in Figures 3A, B and C. Qualitatively the loading curve shows all of the three stages described in the article and in the previous section. Basically, the same observations can be made during the stage IIIB as for the other samples, with densification and thickening of twin lamellae (Figure 3E). Importantly, the present and the previous Spath samples exhibit very similar loading curves, with approximately the same finite strains and flow stresses at each stage. The most striking point is that the sample does not show any evidence of micro-fracturing at any stage of the deformation process. The specimen remains intact and the twinning planes define a well localized deformation band across the whole sample. Though, the flow stresses remain very comparable for both the present and previous samples, which indicates for the latter that the micro-fracturing may not have played a dominant mechanical role.

It has been possible to proceed to an EBSD analysis to get the orientation of the host crystal lattice and also of the twinned part. The Schmid factor calculated using the EBSD data is 0.16. The applied stress at the appearance of the first twin lamella is 29.7 MPa. Accordingly, the CRSS value is calculated at about 5.87 MPa, which is substantially higher (10 times) than the CRSS calculated for synthetic crystals. The differences in terms of sample integrity between different Spath specimens and in terms of CRSS between Spath and synthetic single crystals can easily be related to different initial states. In the former natural Spath, the presence of pre-existing crystal defects (as for instance undetected twins, micro-fluid inclusions, higher dislocation densities…) cannot be excluded.

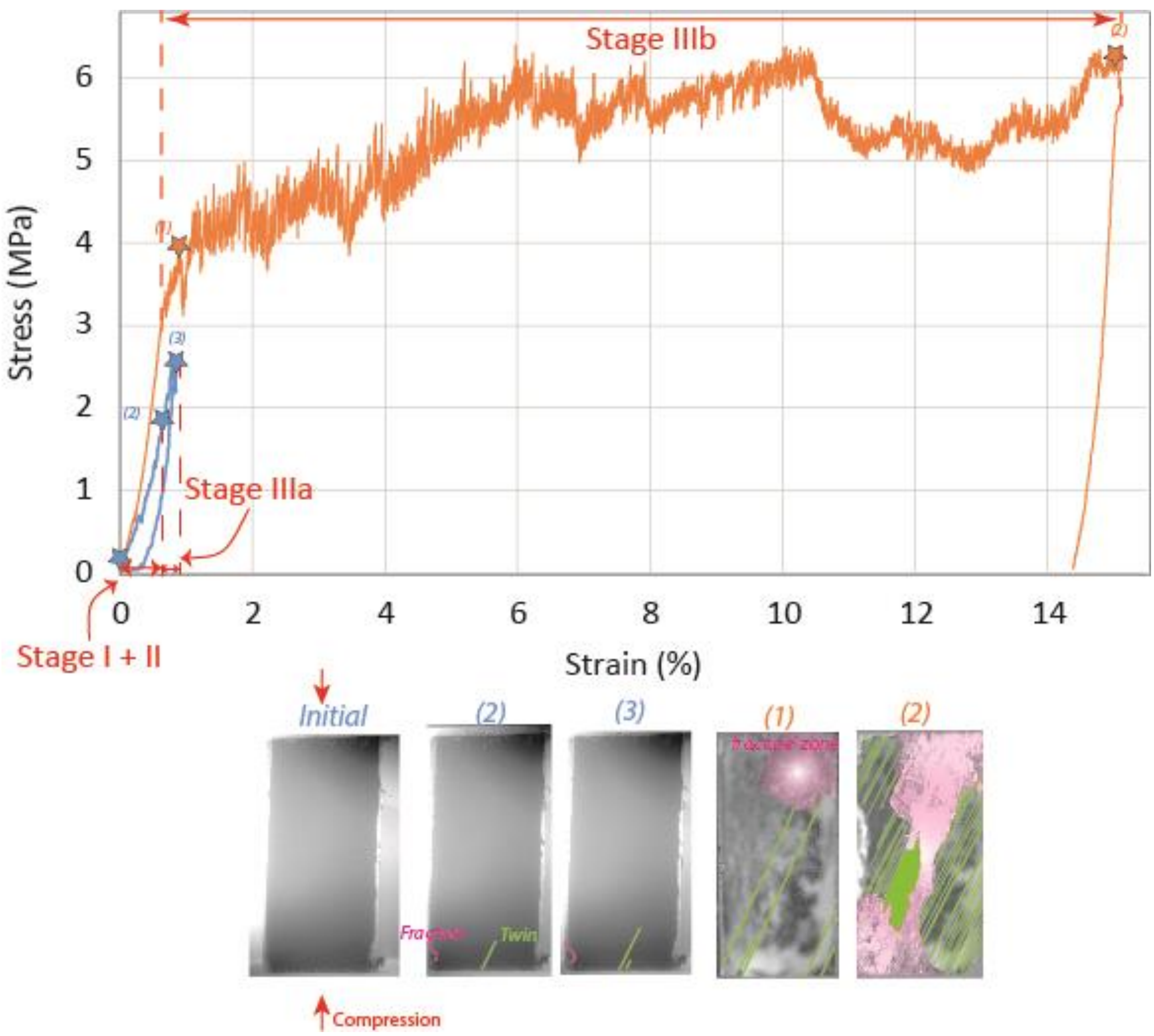

Figure 1: Results from sample 4 x 4 x 8 mm^3 compressed along the direction $[01\bar{1}0]$. Stress (MPa) versus strain (%) curve showing the three stages described in the article. Between the blue and orange curves, the sample has been removed from the press in order to determine the orientation of the first twin lamella. Each star on the curves corresponds to one of the high-resolution pictures below. In the different pictures, the twin lamellae are in light green color. Fractures are in pink color.

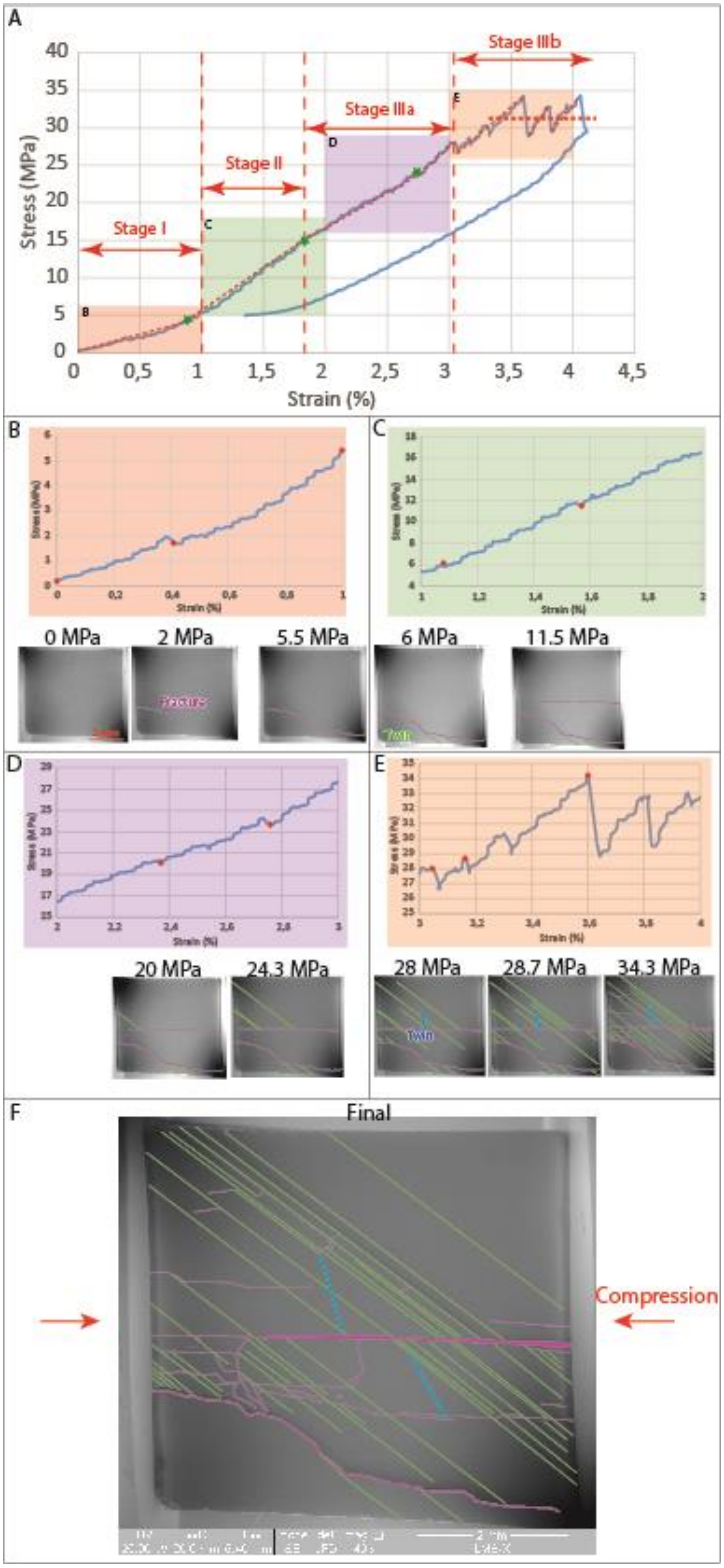

Figure 2: Results from sample Pita. Stress (MPa) versus strain (%) curve showing the three stages described in the article. Each red point on the curves corresponds to one of the high-resolution pictures below. In the different pictures, the twin lamellae are in light green color. Fractures are in pink color.

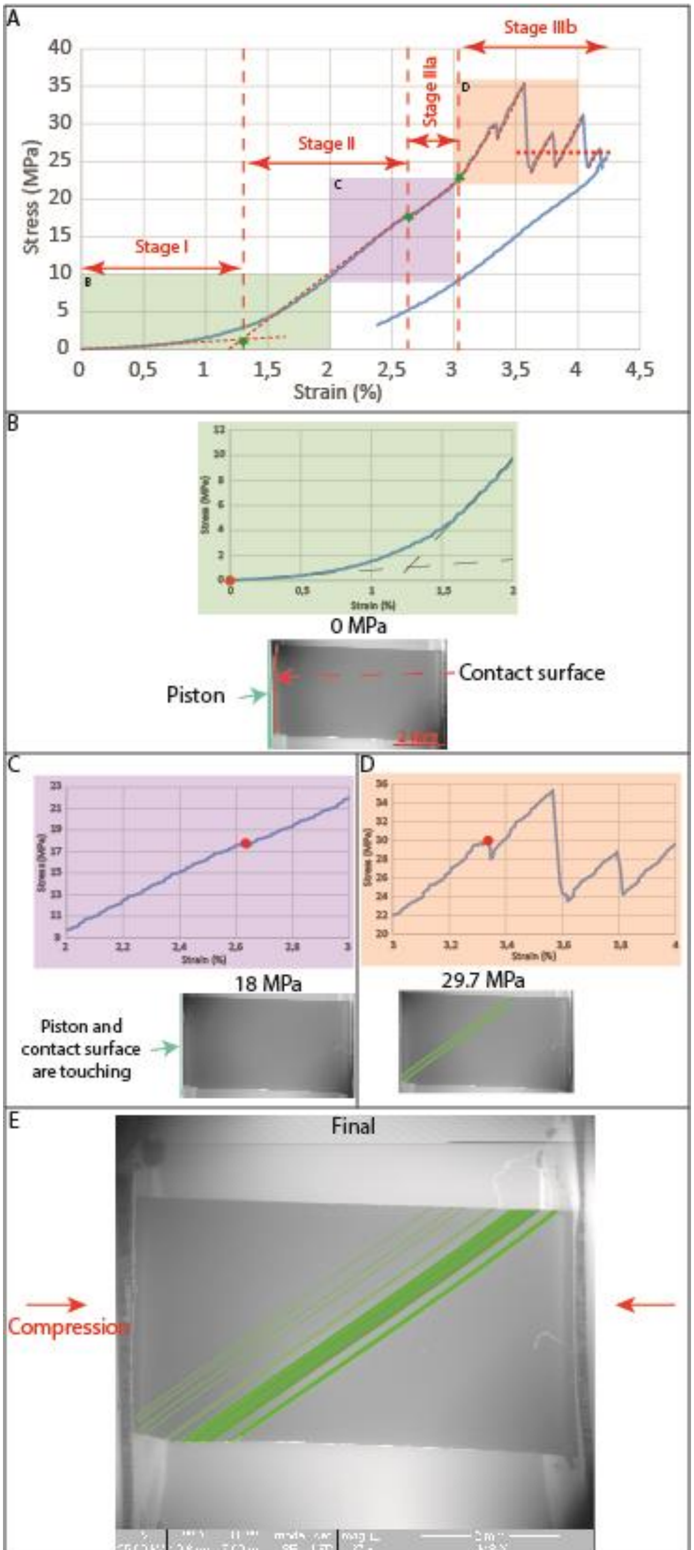

Figure 3: Results from sample Johnny. Stress (MPa) versus strain (%) curve showing the three stages described in the article. Each red point on the curves corresponds to one of the high-resolution pictures below. In the different pictures, the twin lamellae are in light green color. Fractures are in pink color.