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

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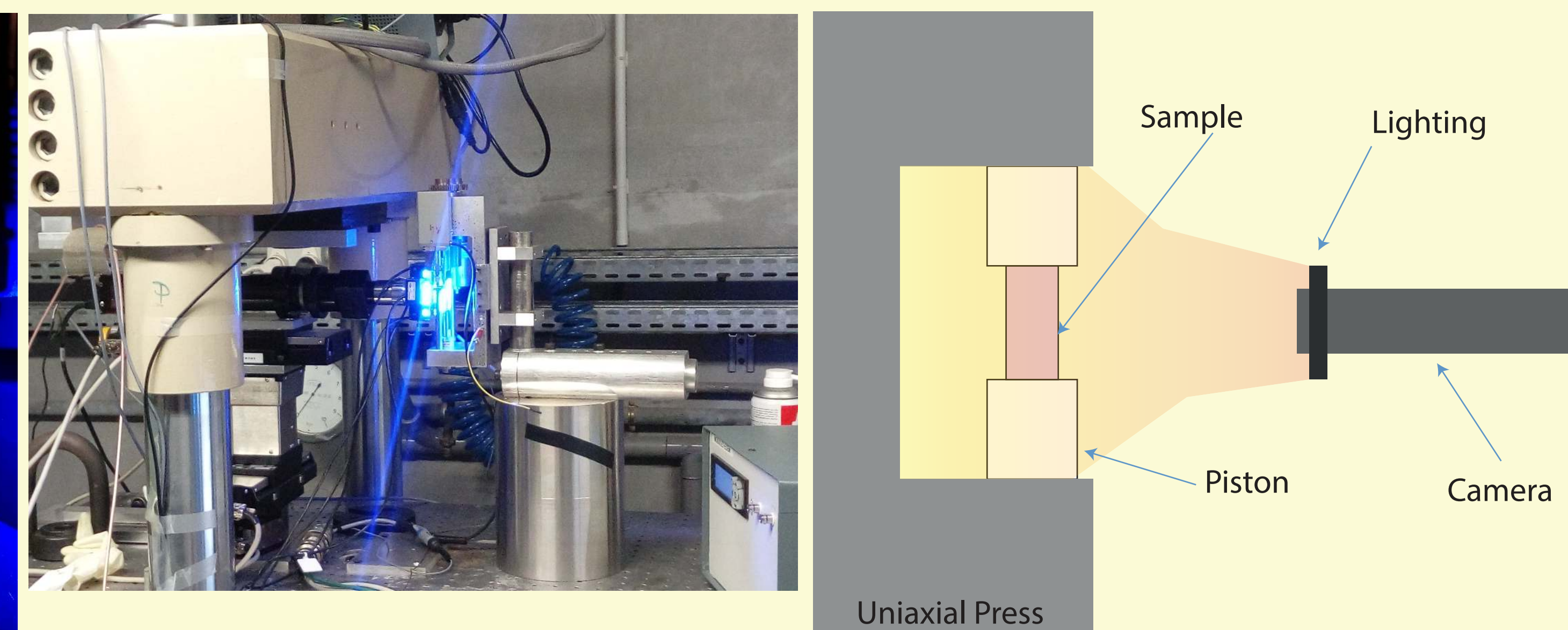
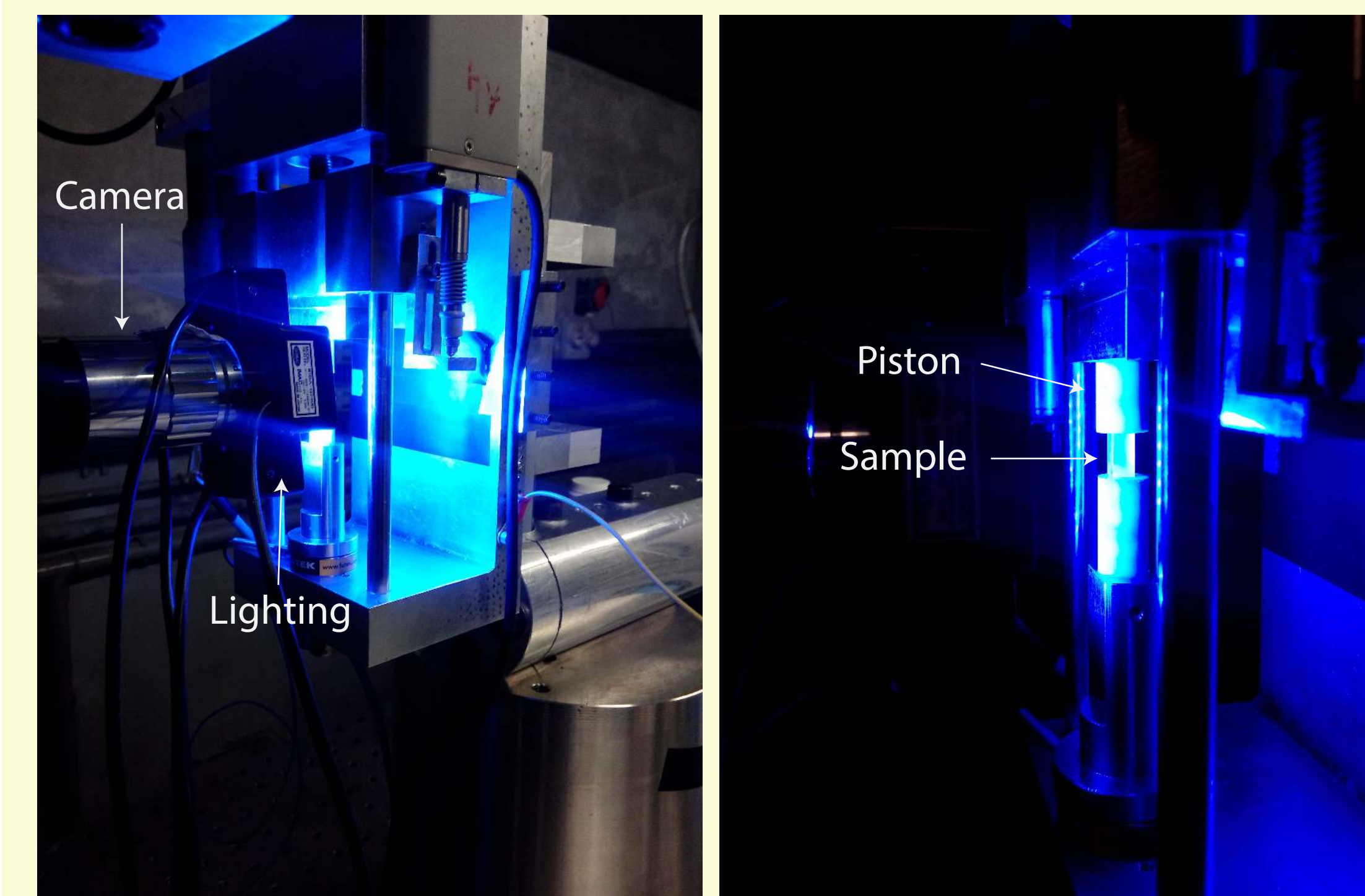
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INTRODUCTION

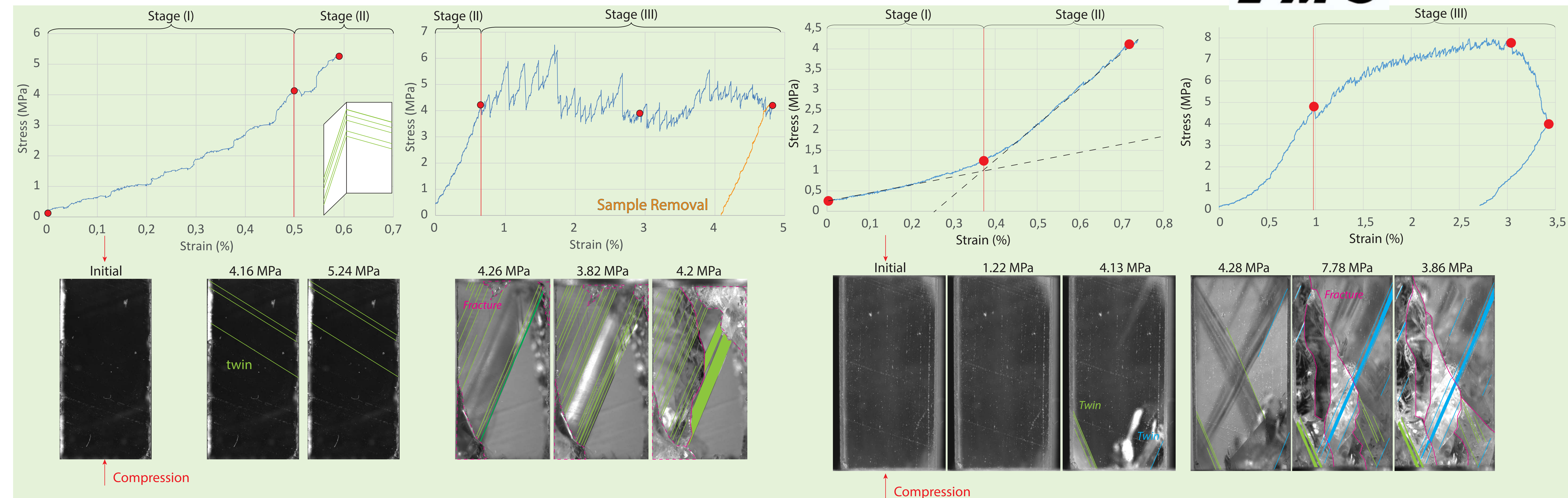
E-twinning is a common plastic deformation mechanism in calcite. Previous experiments have shown that temperature, strain rate and confining pressure have negligible effects on twinning activation, the latter being mainly dependent on differential stresses. Temperature is however reported to control the aspect of twin lamellae, with thickness exceeding 2-5 μm mostly at temperature above 200 $^{\circ}\text{C}$. The critical resolved shear stress (CRSS) required for activation of twinning has been shown to be dependent on grain size and to be subjected to strain hardening: its value increases with the amount of strain accommodated and decreases with increasing grain size. This CRSS value may obey the Hall-Petch relation, but due to sparse experimental data the actual evolution of the CRSS with grain size and strain still remains a matter of debate. Numerous experiments were carried out at high temperature to investigate gliding systems in calcite, but just few data are available on the plastic behavior of calcite crystals at low temperature, despite the fact that the latter may help understand deformation processes of carbonate rocks in sedimentary basins. New mechanical tests were carried out at room temperature on unconfined single crystals of calcite, with different sizes and crystallographic orientations. Uniaxial deformation was performed at controlled displacement rate, meanwhile the sample surface was monitored using optical microscopy (reflected light) and high resolution CCD camera. The retrieved macroscopic stress-strain behavior of the crystals was correlated with the surface observations of the deformation process.

MATERIAL AND METHOD



Scheme and pictures of the experimental equipment used for this study. Only one piston is movable. Its displacement is measured by a LVDT sensor fixed on the frame. The monitoring uses a high-resolution camera equipped with reflection lighting.

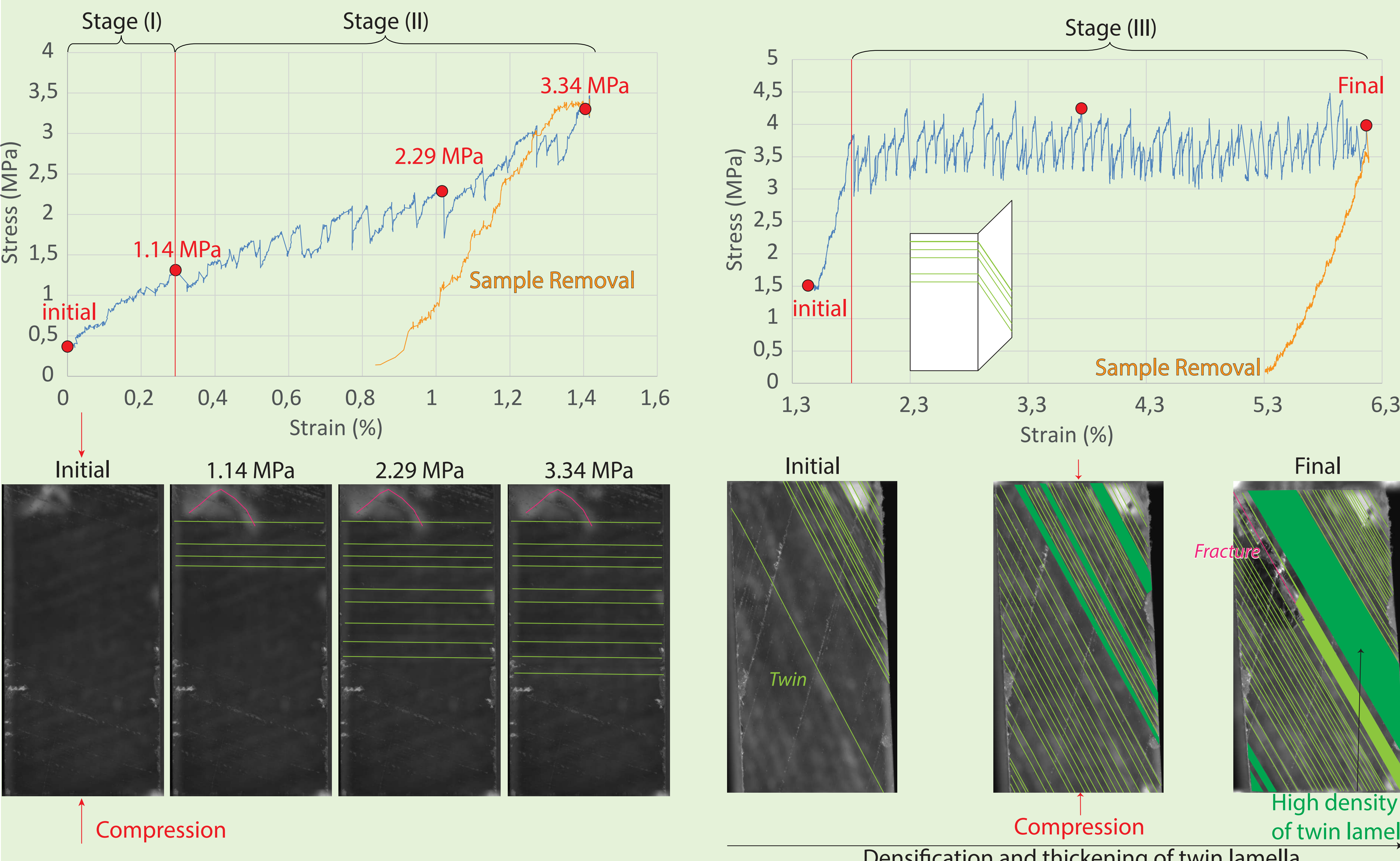
Two grain sizes with two different crystallographic orientations:
 (1) 4 x 4 x 8 mm with the longest axis along [2110] and [0110]
 (2) 3 x 3 x 6 mm with the longest axis along [2110] and [0110]
 Compression is applied along the longest axis and without confining pressure at constant strain rate (10-5 s⁻¹) and room temperature. In order to precisely measure both stress and strain at twinning activation, the experiments are monitored using a Keyence high-resolution camera. When the first lamella appears, the sample is removed from the uniaxial press to measure the twin lamella orientations using an EBSD (Electron Back-Scatter Diffraction). Then, the sample is put back in the press to increase deformation until the sample fails.



Results on the sample 3 x 3 x 6 mm compressed along the direction [2110].

Results on the sample 4 x 4 x 8 mm compressed along the direction [2110]. In the different pictures, the blue and light green colors are used to highlight the presence of twin lamellae of different orientations.

RESULTS

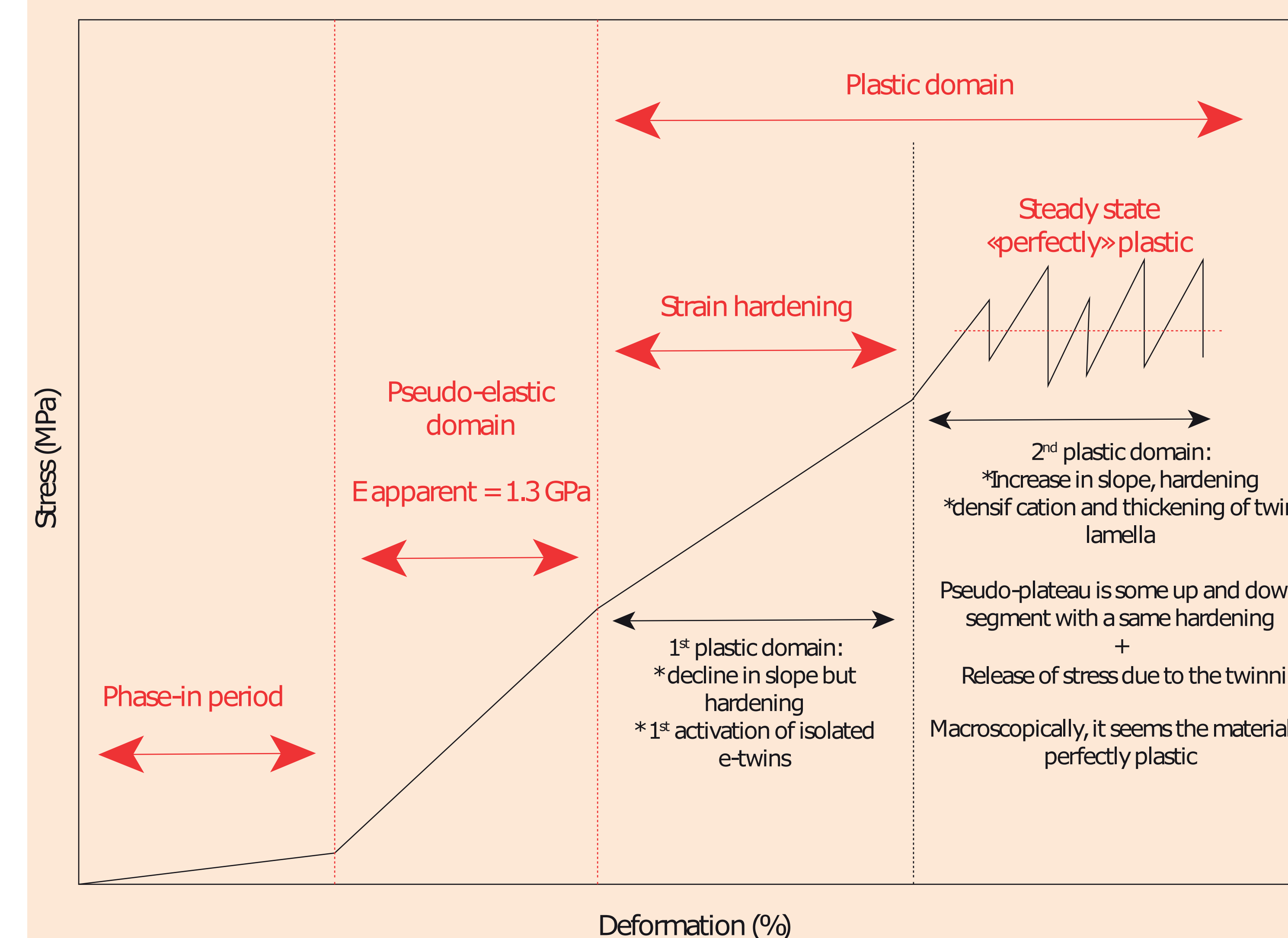


The retrieved macroscopic stress-strain behavior of the crystals was correlated with the surface observations of the deformation process. For all samples, we observed 3 distinctive stages:

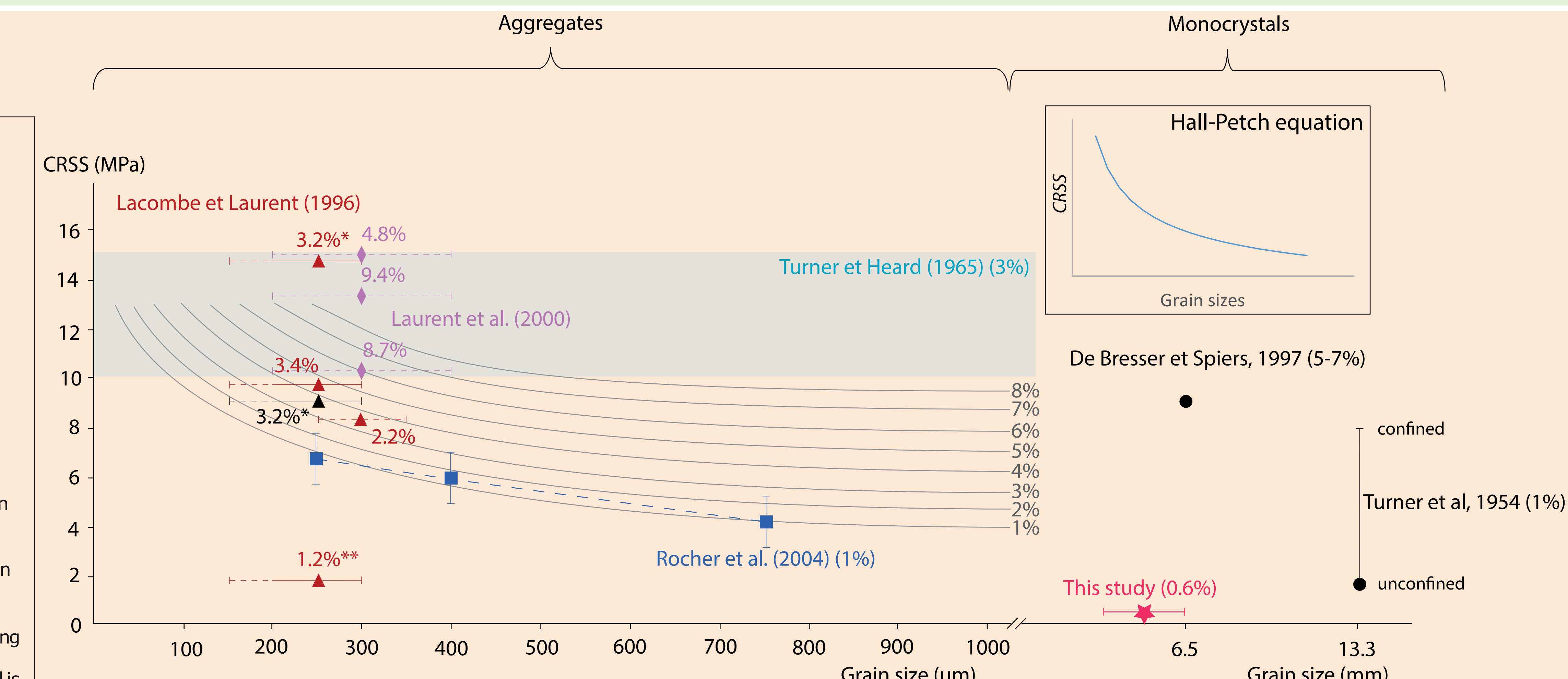
- I: elastic deformation.
- II: onset of plastic deformation characterized by appearance of the very first isolated twins during strain hardening. The lamellae successively appear with increasing deformation, with an average $\sim 2 \mu\text{m}$ thickness.
- III: burst-like densification and thickening of twin lamellae during the final flow stress leveling stage (« plateau »). The latter accounts for most of the irreversible strain and corresponds to episodically fluctuating resistance, with strain hardening increments followed by equivalent stress drops related to twinning events. During this stage, for all experiments, the resolved shear stress values for e-twin activation remain quite steady. Twinning strain is not sufficient to accommodate applied deformation, which leads to micro-fracturing in addition to twinning. Micro-fracturing does not seem to be controlled by the pre-existing twin lamellae.

The first twin lamellae appear at resolved shear stress values of $0.90 \pm 0.35 \text{ MPa}$ during the experiments. This value increases with strain hardening until the «plateau» is reached. Thickening of twin lamellae occurs in 3 steps during stage III:
 (1) Densification of twin lamellae
 (2) Individual thickening of each existing twin lamellae
 (3) Merging of thickened twin lamellae
 For mm-sized single crystals compressed without confining pressure at room temperature, the thickness of a twin lamella can reach 100 – 200 μm for the biggest one.

DISCUSSION AND CONCLUSION



Scheme summarizing observations and interpretations about the macroscopic behaviour of the synthetic monocrystals during uniaxial compression at room temperature.



Summary of the knowledge concerning the evolution of the critical resolved shear stress value (CRSS) with the strain hardening and the grain size.
 *Effect of temperature on the thickness of twin lamellae (Burkhard, 1993): our experiments show thickening of twins at low temperature, in agreement with recent results by Rybacki et al. (2013). However, a strong effect of the absence of any confinement cannot be excluded.
 *Significance of the CRSS: the question about the appropriate value of the CRSS to be considered when using calcite twin data for inversion purposes aiming at retrieving past stress orientations and magnitudes remains unanswered. Should we consider the value at the onset of isolated twin activation, which varies with strain hardening? Or the nearly constant value at the onset of densification and thickening («plateau» during the stage III)?

*Evolution of the critical resolved shear stress (CRSS) value with grain size: we do not see any clear crystal size effect on the CRSS values, but this may be due to the very large size of the crystals. More experiments on both single crystals and aggregates are needed to validate this observation.

REFERENCES

K. Amrouh. 2010. Apport de l'analyse microstructurale à la compréhension des mécanismes de glissement: Exemples de structures plissées aux USA (Wyoming) et en Iran (Zagros). Doctoral thesis, Geology, University of Pierre and Marie Curie, pp. 56.
 M. Burkhard. 1993. Calcite twins, their geometry, appearance and significance as stress-strain markers and indicators of tectonic regime: a review. *Journal of Structural Geology*, 15, 351-368.
 J.H.P. De Bresser, C.J. Spiers. 1997. Strength characteristics of the f, f, and c slip systems in calcite. *Tectonophysics*, 272, 1-23.
 O. Lacombe, P. Laurent. 1996. Determination of deviatoric stress tensors based on inversion of calcite twin data from experimentally deformed monophase samples: preliminary results. *Tectonophysics*, 255, 189-202.
 P. Laurent, H. Kern, O. Lacombe. 2000. Determination of deviatoric stress tensors based on inversion of calcite twin data from experimentally deformed monophase samples. Part II. Axial and triaxial stress experiments. *Tectonophysics*, 327, 131-148.
 C. Parlangeau. 2017. Quantification des paléocroissances par l'analyse des macles de la calcite: nouvelle approche d'acquisition et d'inversion des données et mécanique du maillage. Doctoral thesis, Geology, University of Pierre and Marie Curie, 255-295.
 M. Rocher, M. Cushing, F. Lemeille, Y. Lozac'h, J. Angelier. 2004. Intraplate paleostresses reconstructed with calcite twinning and faulting: improved method and application to the eastern Paris Basin (Lorraine, France). *Tectonophysics*, 387, 1-21.
 E. Rybacki, B. Evans, C. Janssen, R. Wirth, G. Dresen. 2013. Influence of stress, temperature, and strain on calcite twins constrained by deformation experiments. *Tectonophysics*, 601, 20-36.
 F. Turner, D. Griggs, H. Heard. 1954. Experimental deformation of calcite crystals. *Bulletin of the Geological Society of America*, 65, 883-934.