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Indentation unloading phase transformations in silicon: A new perspective

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Indentation Unloading Phase Transformations in Silicon: A New Perspective

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Materials Science

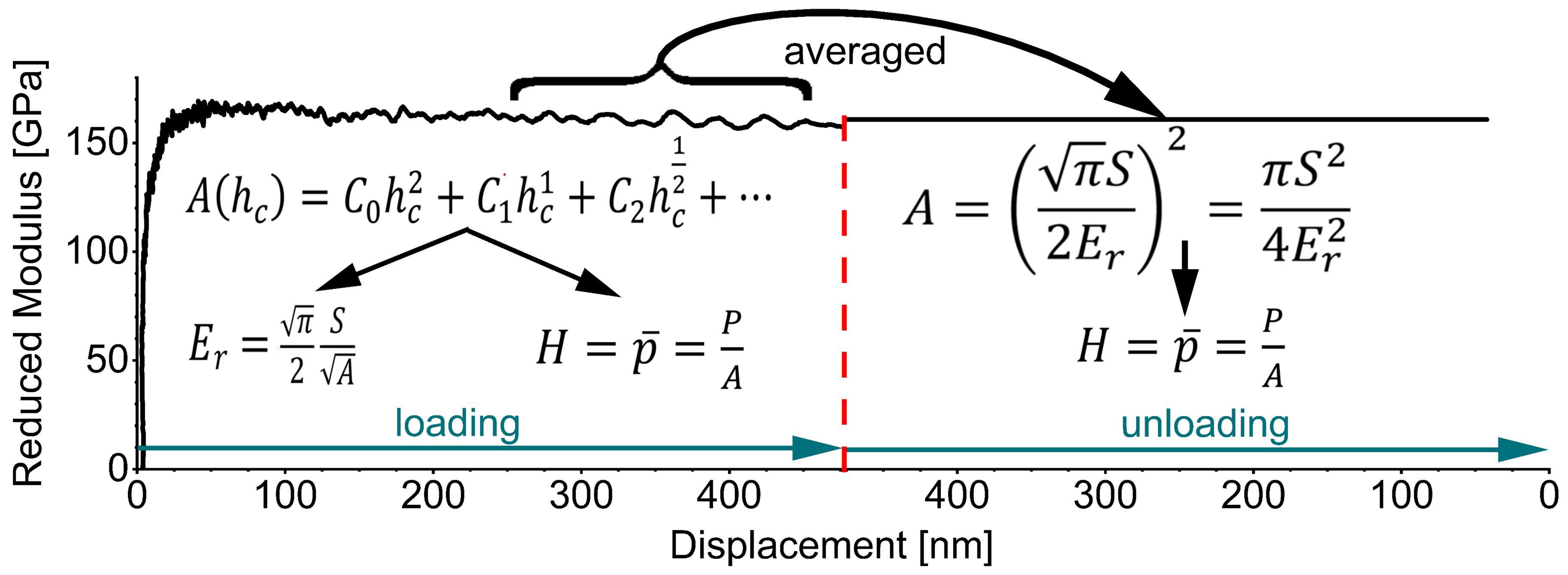
Introduction

The load-displacement curve shows abnormalities during the unloading of indentations in silicon. These abnormalities are linked to phase transformations [1]. Applying dynamic nanoindentation to the unloading segment should, in theory, allow the evaluation of the contact pressures at which such transformations occur. However, these methods are typically designed to only measure mechanical properties during the loading process [2]. The nanoindentation protocol presented in this work aims to make the transformation pressures directly accessible [3]. Furthermore, constant-load holding segments were added during unloading. The impact of such segments on the phase transformations was assessed with Raman spectroscopy.

Symbols/Abbreviations

P	load	E_r	reduced elastic modulus	**CSM**	continuous stiffness measurement
H_c	contact depth	S	dynamic contact stiffness		
A	contact area	C_i	area function fitting constants		
H	hardness	$\bar{p}$	mean contact pressure		

Methodology

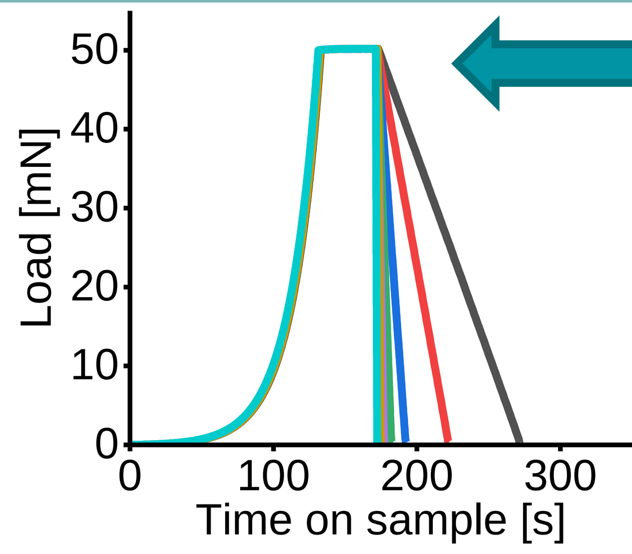


Schematic of the usage of the reduced elastic modulus in the new indentation protocol used to measure the transformation pressures [3]. The area function of the tip holds during loading, and together with the CSM signal, the mechanical properties can be calculated with the Oliver-Pharr formulas [4]. However, during unloading, the area function loses its validity [2].
The approach is now to determine an average reduced elastic modulus during loading. This value is then used to calculate a contact area during unloading. Subsequently, an "unloading hardness" (the mean contact pressure) can be calculated. [3]

Summary

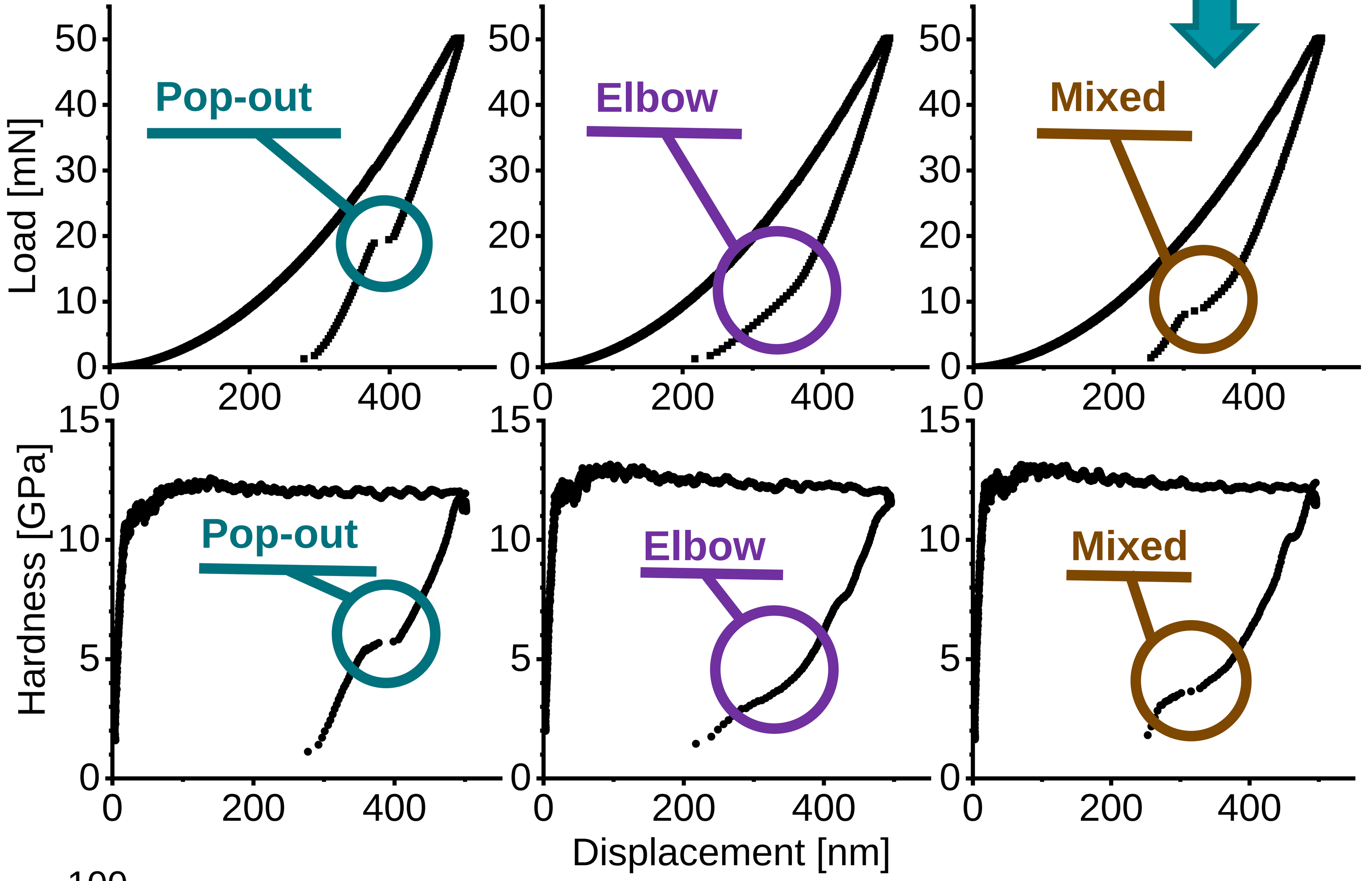
- Under the assumption of a constant reduced modulus, CSM can be used to calculate the contact pressure during unloading uninterruptedly.
- Continuous unloading experiments show that the transformation pressures are lowered for faster unloading rates.
- Transformations also happen purely time-dependent if the contact pressure (and thus the driving force) is kept constant.
- During this holding process, a continuous amorphous transformation occurs.
- High transformation pressures favor the formation of metastable, crystalline silicon, whereas low transformation pressures lead to amorphous silicon.

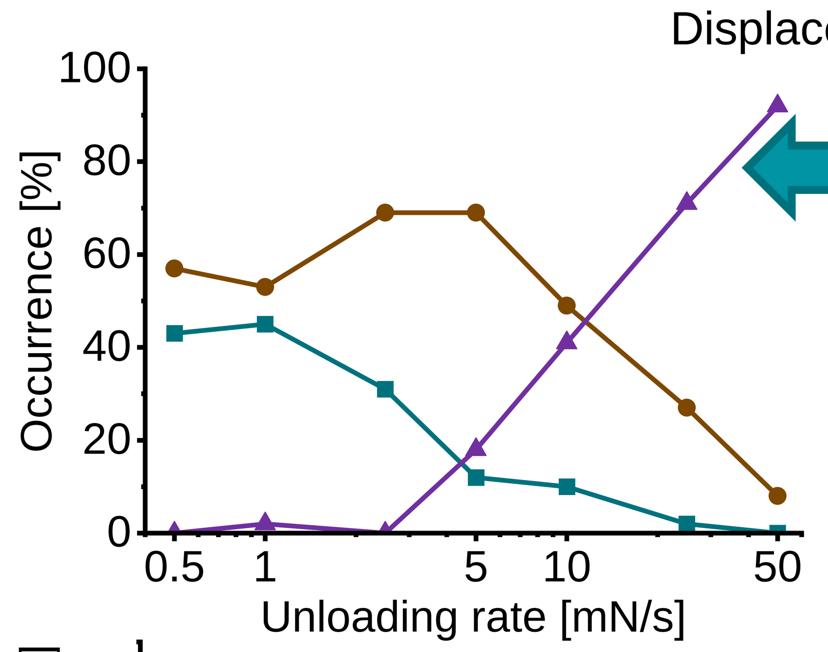
Continuous Unloading



The load profiles used for continuous unloading.

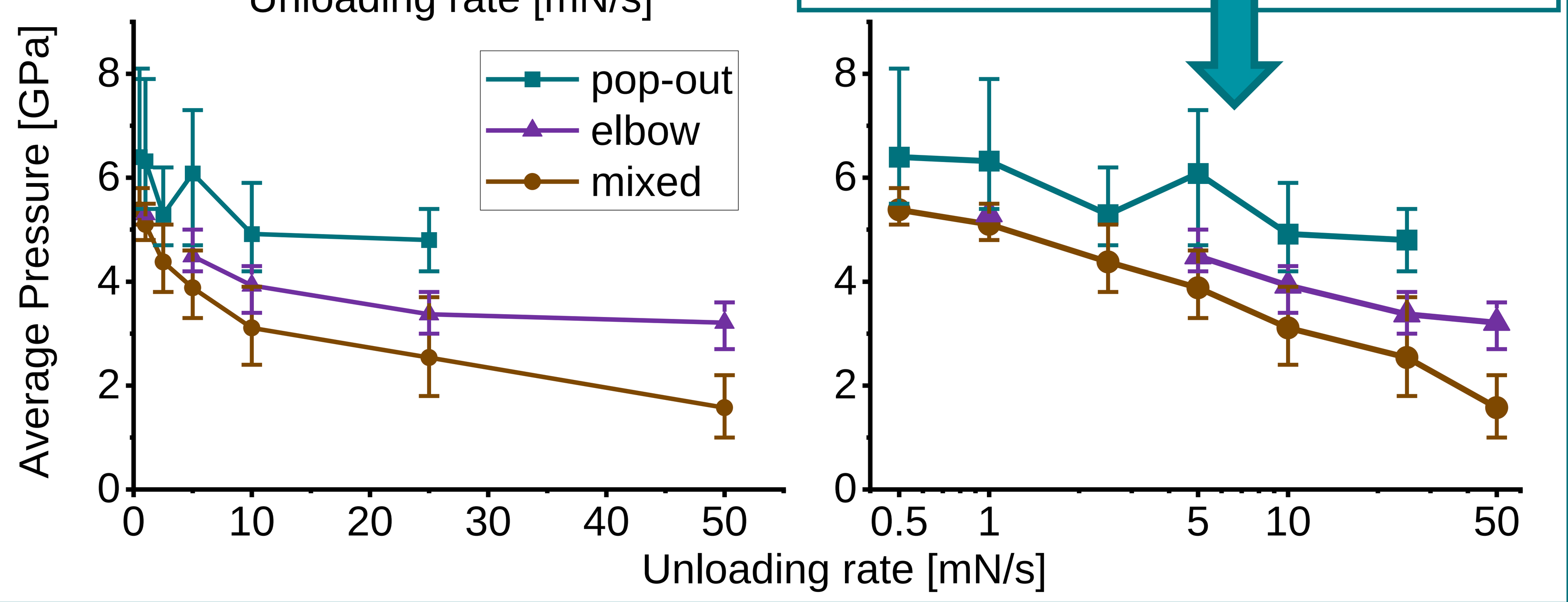
The burst-like pop-out is associated with the generation of metastable, crystalline silicon phases, whereas the continuous elbow leads to amorphous silicon. Transformations to a mix of crystalline and amorphous silicon also lead to a mixed load-displacement response. [1]
The lower line shows representative contact pressure profiles acquired with the new protocol.



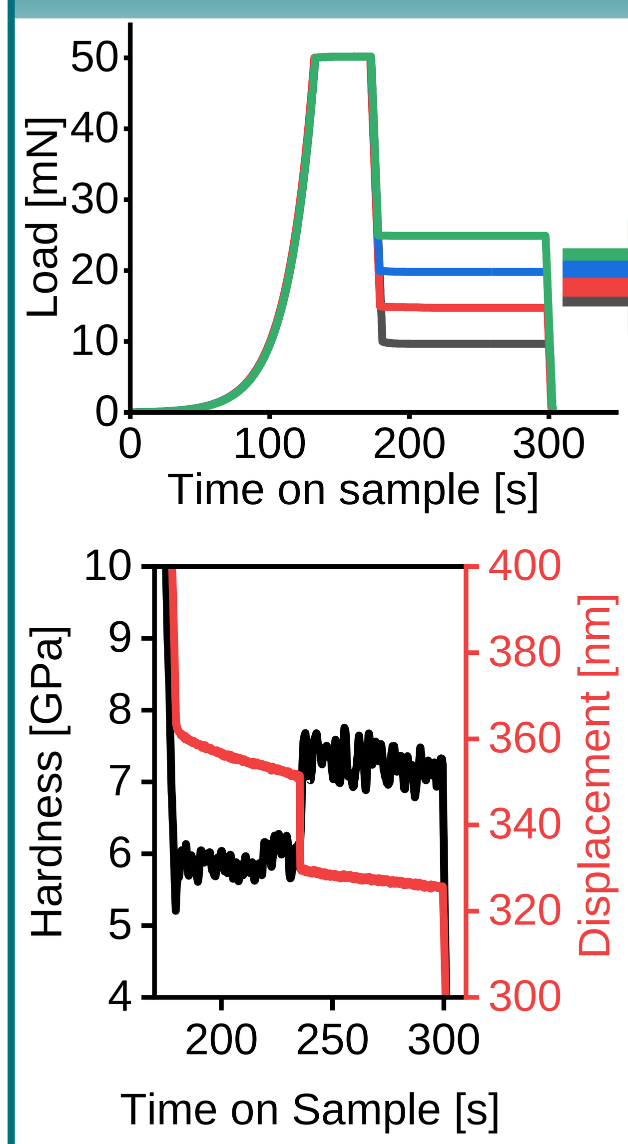


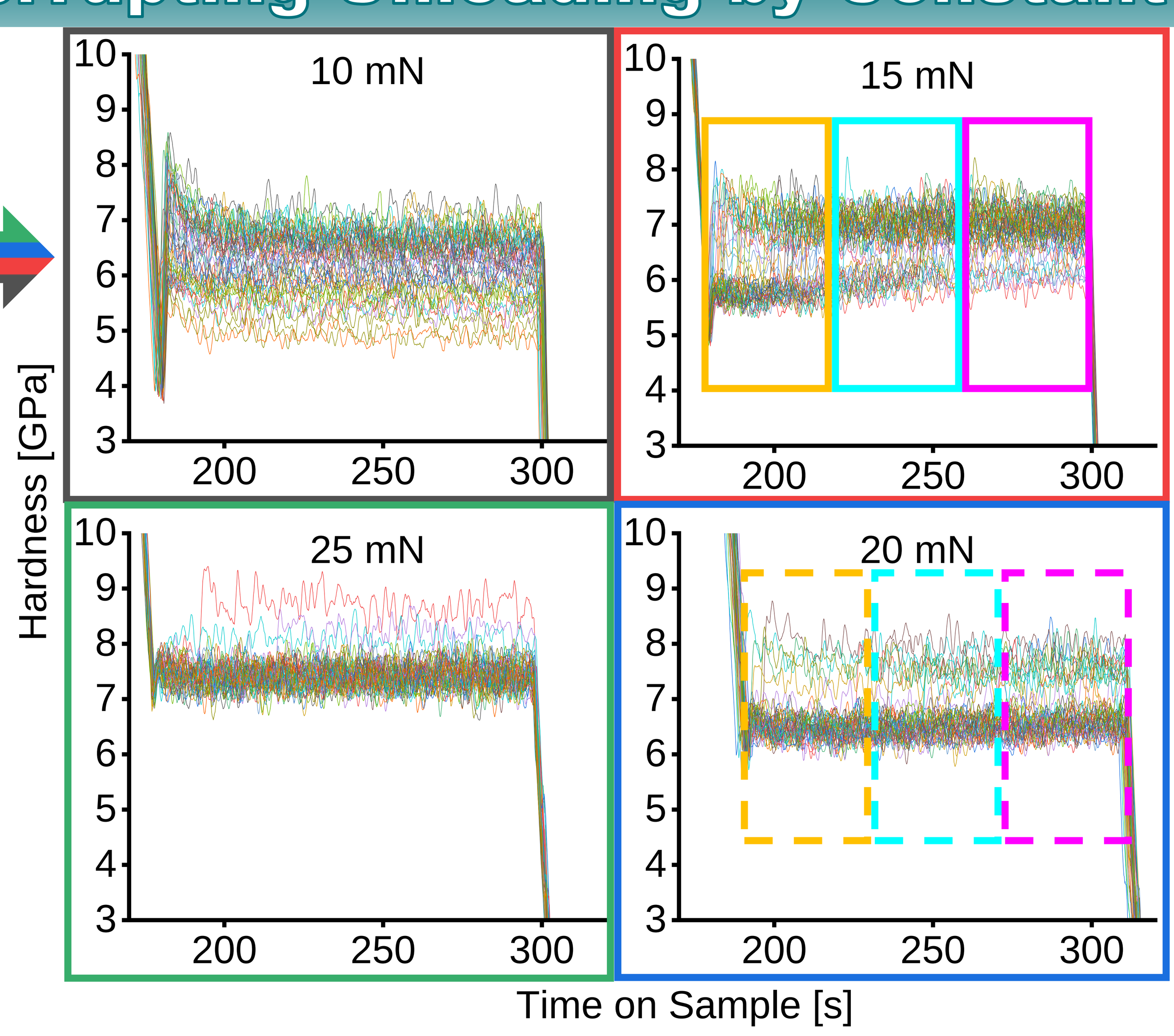
The probability for the different unloading transformations to occur; depending on the unloading rate.

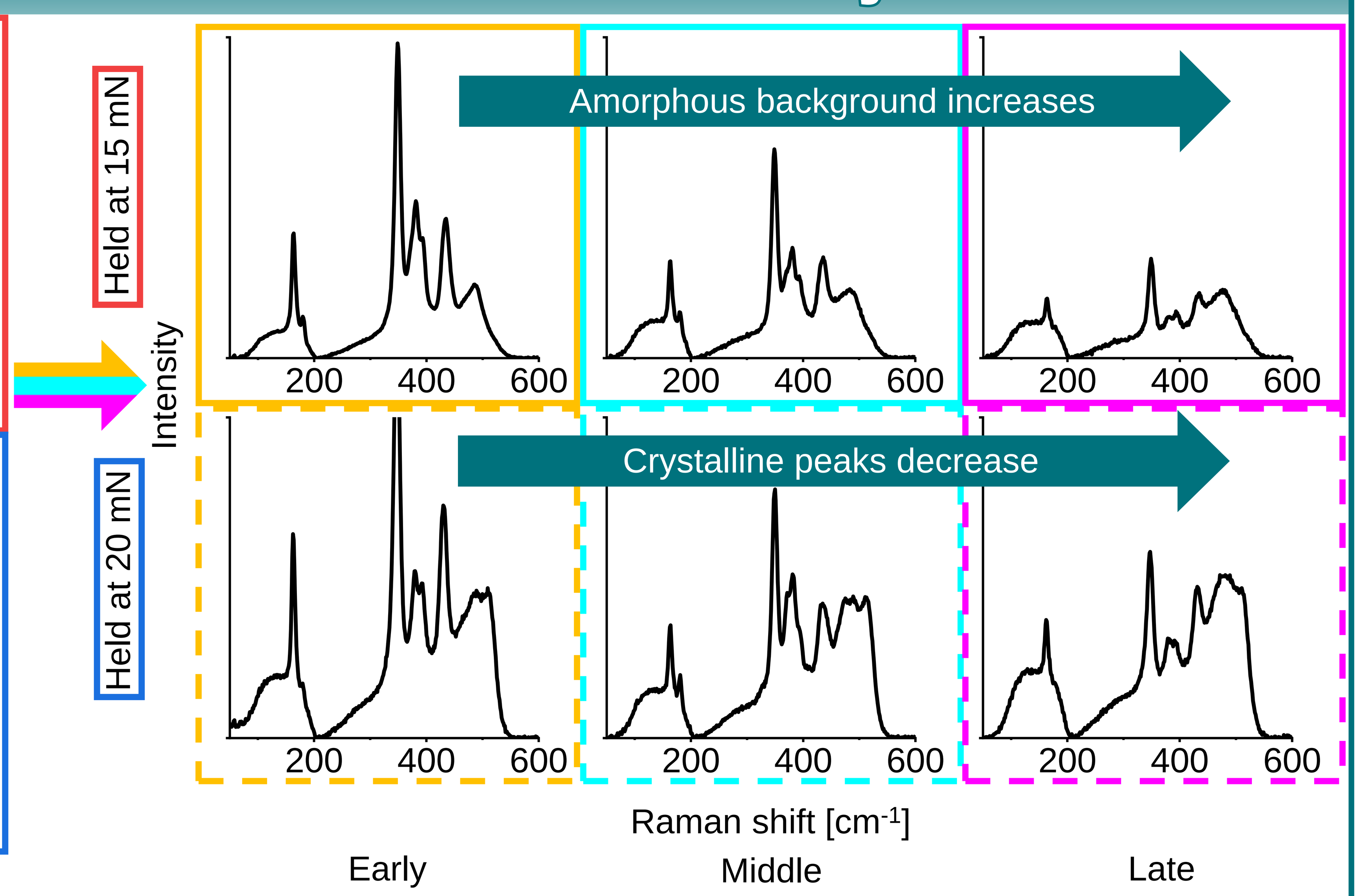
Measured transformation pressures at different unloading rates: Faster unloading delays the transformations to lower pressures (higher driving forces). The error markers indicate the highest/lowest individual value measured.



Interrupting Unloading by Constant Load Holds + Raman Analysis







Above: used load profiles
Below: pop-outs (drops of displacement) while holding cause a contact pressure (hardness) increase

Pressure traces while holding: jumps of the contact pressure to the "upper band" indicate a constant load pop-out. Especially for the 15 mN hold, a continuous increase in pressure can be seen for samples that have not exhibited a pop-out yet.

Averaged Raman Spectra of indents after the holding experiments: Early, middle, and late specify when the pop-outs occurred (within the first, middle, or last 40 s of holding, respectively). The continuous increase in contact pressure can be linked to the generation of amorphous silicon (broad Raman bands), whereas the pop-out leads to metastable, crystalline silicon (sharp peaks).

WHERE RESEARCH MEETS THE FUTURE

[1] V. Domnich et al., *Appl. Phys. Lett.*, vol. 76, no. 16, (2000) [2] B. Merle et al., J. Mater. Res., vol. 27, no. 1, (2012) [3] G. J. K. Schaffar et al., *JOM*, vol. 74, no. 6, (2022) [4] W. C. Oliver and G. M. Pharr, *J. Mater. Res.*, vol. 7, no. 6, (1992)