Constant pressure creep

Long term creep testing:

Long term creep experiments are used on nanoindentation test equipment to study the deformation behavior of materials at low strain rates (10^{-4} – 10^{-6} s^{-1}) commonly not accessible in strain rate controlled loading experiments. When the peak hold segment is extended from seconds to hours, the influence of thermal drift is difficult to account for. Therefore the displacement sensor is often not the best source to measure the slow displacement changes. Instead the utilization of a dynamic modulation of the force and the continuous determination of the dynamic contact stiffness allows to calculate the contact area and therefore the contact depth [1].
Typically, after the loading to a target depth or load, the load on the sample or the raw load is held constant and the displacement is monitored over many minutes. During the constant load period, the indenter (typically Berkovich type) is creeping into the sample, the contact area increases, the mean pressure under the indenter decreases, the contact is “relaxing” while the creep is monitored.

The constant pressure creep method implemented and shown here is based on the idea and publication by Prof. Durst and his group [2].

The approach uses the basic equation, which connects the contact stiffness S_c with the reduced modulus E_r and the contact area A. The geometry factor β is set to 1 according to ISO.

$$S_c = \beta \frac{2}{\sqrt{\pi}} E_r \sqrt{A(h_c)}$$

The dependency $A(h_c)$ describes the commonly used area function, which is calculated from the contact depth h_c. If the reduced modulus is well known and constant, the above equation can be used to calculate the contact area directly from the contact stiffness.

$$H = \frac{F_{max}}{A(h_c)}$$

The above equation for the hardness describes the maximum load that the sample can hold before it yields, but it can also describe the contact pressure for loads, that are smaller than the maximum load. If the area is calculated from the contact stiffness, one can define:

$$H(S_c) = \frac{Load\ on\ Sample}{A(S_c)} = \frac{Load\ on\ Sample}{S_c^2} \cdot 4 \frac{\beta^2}{\pi} \cdot E_r^2$$

This hardness or pressure scales with the square of the reduced modulus and is constant, if the quantity $S^2/Load$ is controlled to be constant.

[1] V. Maier, B. Merle, M.Göken, and K. Durst, J. Mater. Res. 28, 1177 (2013)
[2] O. Prach, C. Minnert, K.E. Johanns, K. Durst, J. Mater. Res. 34, 2492 (2019)

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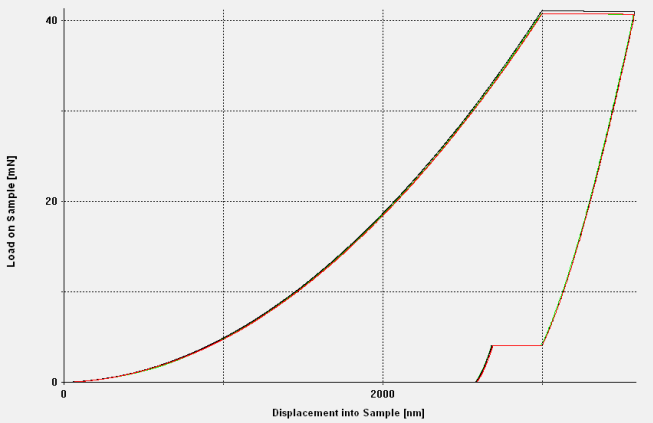
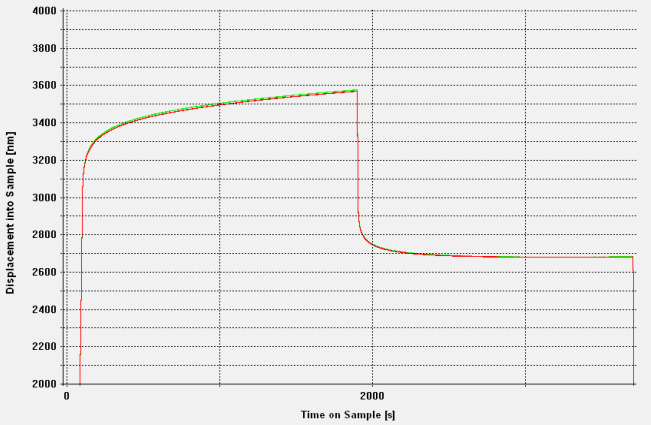
Creep testing SAN for 1800 seconds:

The best fitting E_r value, the maximum hardness and the amount of hardness drop during the creep time should be characterized for every new sample prior to run any constant pressure creep tests.

The creep tests below are performed on a styrol acryl nitril (SAN) polymer sample with significant creep and relaxation. A Berkovich type indenter is loaded until 3000 nm of displacement using a strain rate of 0.05 s^{-1}. The peak load is held constant for 1800 seconds, followed by 1800 seconds of drift determination at 10% of the maximum load.



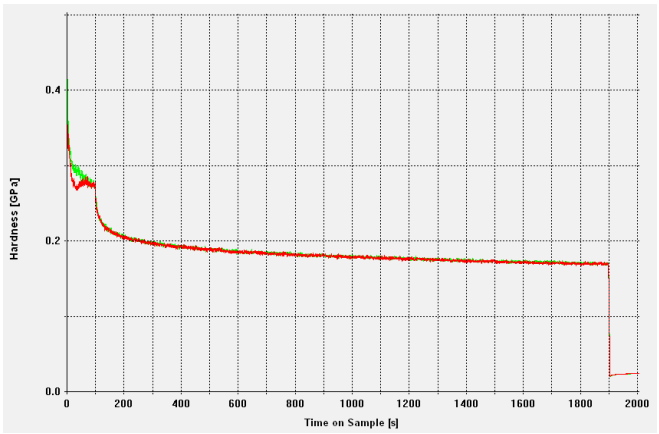
Load vs. displacement into sample



Displacement vs. time on sample

The peak hold at constant load shows a fast initial creep, with maximum velocity at the end of the loading at 3000 nm of displacement. After unloading to 10 % of the maximum load, the polymer shows a pronounced relaxation towards the surface. At the end of the drift determination segment we get reasonable low drift rates like 0.013 nm/s.

The hardness vs. time plot indicates approx. 0.28 GPa during loading, then the pressure decreases to 0.17 GPa during peak hold time. The reduced modulus was evaluated to be stable at 4.8 GPa. For the constant pressure test we used this value for scaling and a pressure target value of 0.2 GPa.



Hardness vs. time during loading (0 - 100 s) and peak hold (100 - 1900 s)

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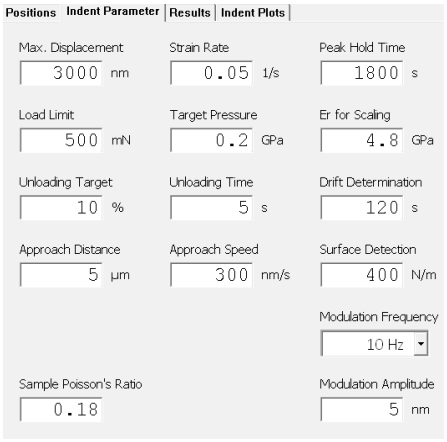
Creep test at constant pressure:

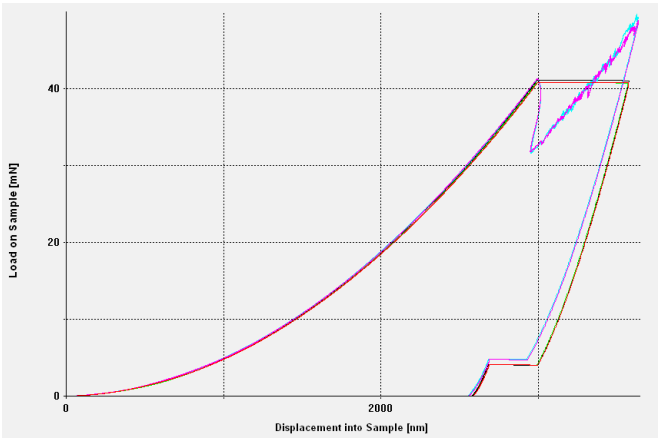
The constant pressure creep method is based on the dynamic testing recipe, where the indenter is pushed strain-rate-load-controlled into the sample, until a maximum load or displacement is reached. In the following peak hold segment the load is not constant, instead a load controller is used to set and maintain a constant pressure level. The inputs for the test recipe are shown below, with a “Peak Hold Time” (creep time) of 1800 seconds. The two inputs below “Target Pressure” and “Er for Scaling” are essential for the constant pressure load controller.

The reduced modulus determination is based on the indenter tip area calibration. It is very important, that the indenter area function is well calibrated before the value “Er for Scaling” is determined.
The target pressure must be lower than the previously determined maximum hardness, otherwise the indenter will simply be pushed into the material.

When the constant pressure control is active, the current load target is calculated based on the current dynamic contact stiffness, target pressure and E_r used for scaling. The contact stiffness is low-pass filtered before the target pressure is calculated to remove high frequency noise inputs to the load target. Differences between actual load and target load are minimized with a proportional controller feedback. Additionally the change of the load is limited by a force rate limit (default:1 mN/s). The controller will end the constant pressure period before the peak hold time has elapsed, when the “Load Limit” is passed, or the load falls below a lower limit.



The parameter inputs for the constant contact pressure test recipe. The new inputs “Target Pressure” and “Er for Scaling” are used in the peak hold segment.



Load vs. displacement into sample comparing the constant load tests to the constant pressure tests. The feedback reduces the load quickly in order to establish the target pressure. The indenter creeps under the target pressure, consequently the feedback increases the load to maintain the target pressure.

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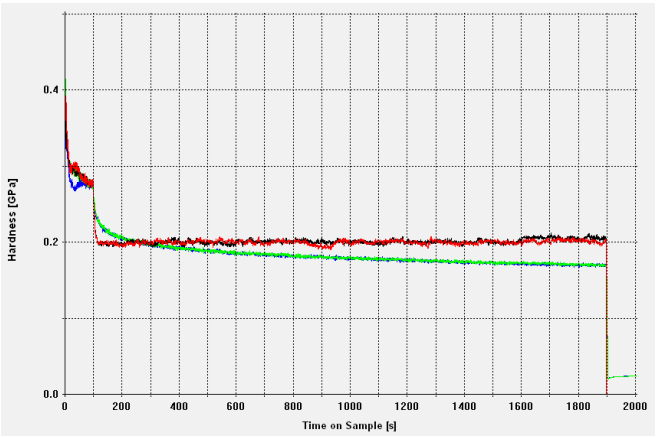
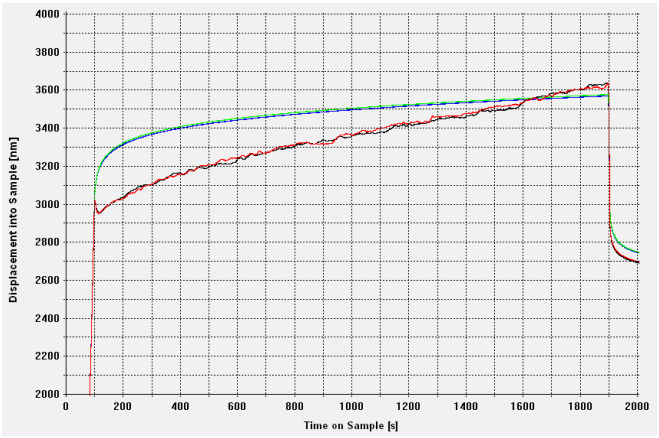
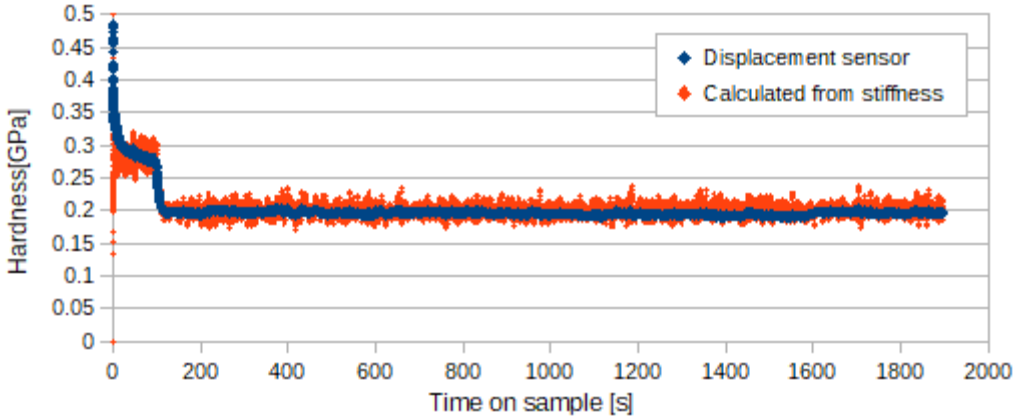
Creep test at constant pressure:



Hardness vs. time on sample. When using the constant pressure feedback, the target value of 0.2 GPa is reached and stable in a few seconds. The hardness values in this graph are calculated based on the displacement reading and tip area function, while in the background the controller is using the contact stiffness and E_r for calculations. Manual drift correction with 0.04 nm/s is applied to the displacement.



Displacement vs. time comparing constant load (green, blue) and constant pressure (red, black). When the load is reduced in order to establish the target pressure, the displacement is slightly reduced, then the displacement increase is initially slower compared to the constant load curves.



The comparison between hardness data calculated from the displacement sensor data and hardness calculated from the dynamic contact stiffness. While the hardness data from stiffness are more noisy, they show a good correspondence to the displacement sensor hardness. This matching is stable over time when using a constant drift rate of 0.04 nm/s. Without drift correction, the hardness from the displacement sensor has a slightly negative slope.

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Calculating the displacement from the stiffness:

For the real-time load feedback the controller is calculating the contact area from the filtered dynamic contact stiffness data. The analysis software also calculates the area from the (unfiltered) dynamic contact stiffness for each data point in the creep segment:

$$A(S_C)=\frac{\pi}{4\beta^2 E_r^2}S_c^2$$

The contact area is connected to the contact depth h_c via the area function:

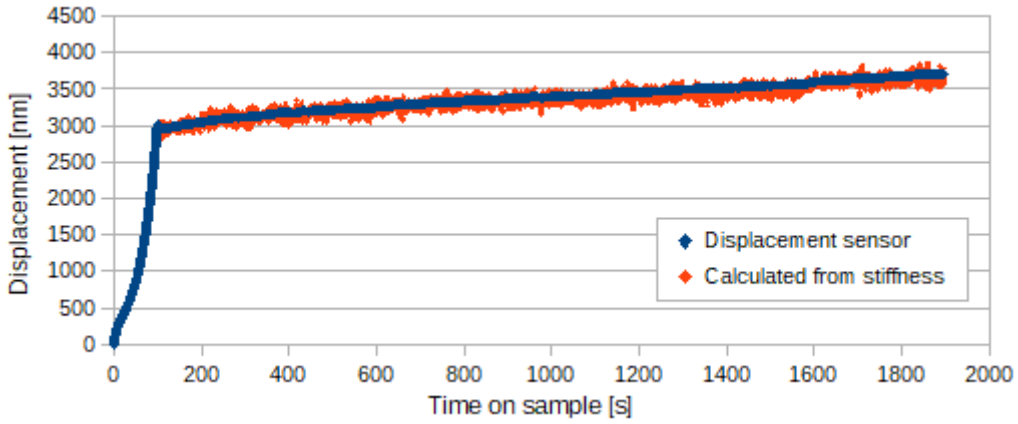
$$A(h_c)=A_0 h_c^2+A_1 h_c+A_2 h_c^{1/2}+A_3 h_c^{1/4}+\ldots$$

For each $A(S_c)$ we can numerically find the corresponding $h_c(S_c)$.

Finally we calculate the displacement into sample using:

$$h(S_c)=h_c(S_c)+\varepsilon\frac{Load\ on\ sample}{S_c}$$

The calculated values for “Hardness from Stiffness” and “Displacement from Stiffness” are accessible when exporting the data in the analysis tool.



Comparison between the displacement directly from the displacement sensor data and the displacement calculated from the dynamic contact stiffness. During the loading segment, both curves are on top of each other.

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