BRUKER

Application Note #1513

Time-Dependent Deformation Behavior of PMMA

Time-dependent deformation of materials is typically not investigated by hardness/modulus measurements. To ensure comparability of measurements, load functions must be appropriately matched. There are many different material cases where the effects of a changing time scale produce negligible hardness changes and, therefore, testing at a constant strain rate is not a primary concern. The first observations of time-dependent deformation behavior, with a corresponding concept for its analysis, was observed on low-melting-point metals, which exhibit a significant amount of creep.[1] Other metals that exhibit creep at room temperature (RT) include aluminum and copper. Constant strain rate tests are becoming increasingly important for other metals as well due to recent innovations in high-temperature testing where their melting point is being approached. Another class of materials whose creep deformation is under investigation is polymers. Here, the time dependency in their properties can be described by their viscoelastic behavior, which can be tested in a nanoDMA® III experiment or through measurement of creep rates (for those polymers that show a low-loss). Creep rates are typically used to characterize material behavior at very small deformation speeds. While the creep rate monitors the deformation velocity of a material with respect to the applied stress, a constant strain rate experiment aims to control the deformation speed and measure the necessary pressure. Constant strain rate experiments with an indenter are typically performed in a strain rate range of 1 to $0.001s^{-1}$, while creep experiments explore a strain rate range of 0.001 to $0.000001s^{-1}$.[2]
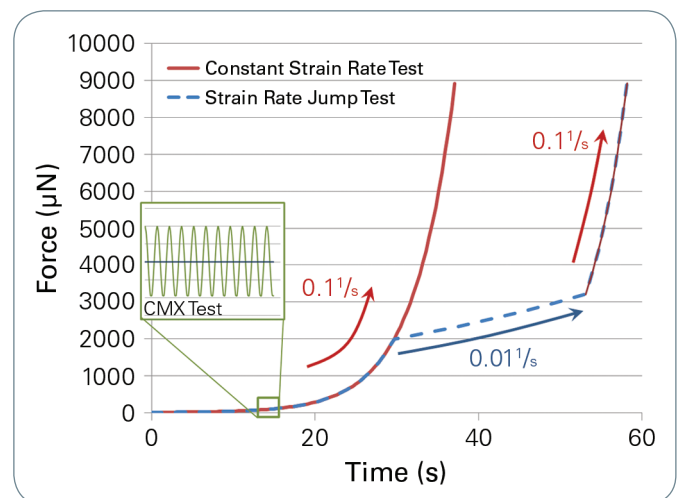

Figure 1. CMX load function with a constant strain rate profile. Red portions are performed at loading rates of $0.1^{1}/s$. The blue load function jumps between a loading rate of $0.1^{1}/s$ and $0.01^{1}/s$.

Indentation Strain Rate Definition

In a tensile test, the strain rate is defined as the ratio of the deformation velocity, dl/dt, to the length of the tensile sample.[1] In an indentation experiment, it is inappropriate to relate the deformation speed to the sample size. The most straightforward length scale available is the penetration depth of the indenter, h_c. The strain rate, $d\varepsilon/dt$, is then defined as $dh_c/dt/h_c$, though it should be noted the applied stress state is not strictly comparable to a tensile test. Since the indentation depth, h_c, is always very small during the initial stages of testing, it becomes clear that the strain rate can be very high. Assuming a sample with constant hardness, it was shown that a constant strain rate experiment could be conducted with a Berkovich indenter.[1] Under these conditions, a strain rate $dh_c/dt/h_c =$ const. would be fulfilled if the force, P, would change in a similar way, $dP/dt/P =$ const. This requires an exponential load function for the indentation. Recently, Meyer et al. have shown the effects of a change in strain rate on nanocrystalline Al during an indentation experiment.[4] They showed that the stress level necessary to achieve a certain strain rate is represented by the hardness, i.e., the average pressure under the indenter, which changes in response to a change in strain rate specified by the load function. Furthermore, some time is needed at a given strain rate to achieve a steady-state hardness measurement. For the present study, similar experiments have been conducted on PMMA, a glassy polymer, at RT. Below, we compare the constant strain rate experiments with strain rate jump tests and creep studies for the very low strain rate values.

Indentation Testing on PMMA

Indentation experiments were carried out on a PMMA sample with different strain rates (0.1, 0.01, and 0.001s^{-1}) and by strain rate jump experiments. The strain rate jump experiments started at a strain rate of 0.1s^{-1}. At a load of 1900 μN, the strain rate was changed to 0.01 and 0.001s^{-1}. Additionally, a reference creep test was performed over a time frame of 8000s in order to study the creep rate of PMMA at strain rates between 0.001 and 0.00001s^{-1}. The creep experiment was performed after a fast loading of the indenter to 9000 μN. The modulus shown in Figure 3 appropriately does not depend on the penetration depth or on the strain rate used for the experiment, as it does not relate to the plasticity of the sample. This is an expected result and provides verification of applied test methodology. The average pressure under the indenter can be plotted for the different tests conducted in this study. A linear behavior is found when plotted against the log-scale of the strain rate (Figure 4).
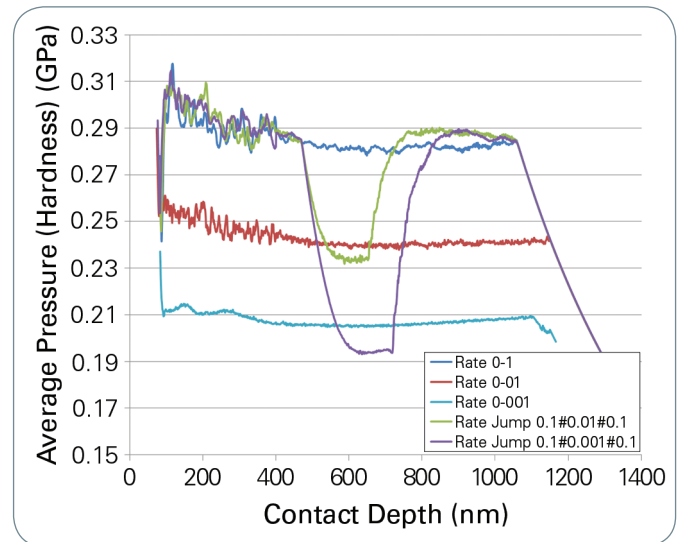

Figure 2. Shows the hardness of the PMMA at different strain rates, it represents the average pressure between the indenter and the sample. It can be clearly seen that during the tests, the stress level for the jump tests changes to the respective constant strain rate. The curves represented here are the average curves of four indentation experiments, which also indicate the reproducibility of the indentation tests.
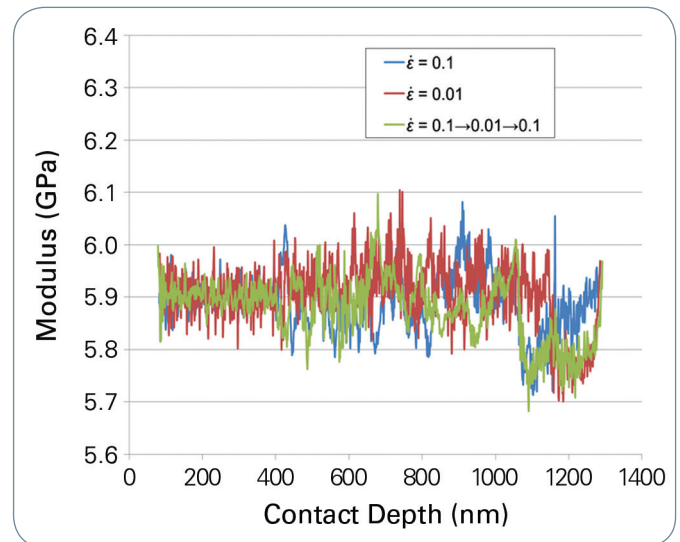

Figure 3. Reduced modulus for the constant strain rate experiment and the strain rate jump test.

Conclusions

Three different tests were performed to study the strain rate behavior of PMMA using a Hysitron® TI 950 TriboIndenter® equipped with nanoDMA III. Figure 4 summarizes the results found by constant strain rate indentation, strain rate jump testing, and by creep testing. These findings allow for the characterization of the PMMA strain rate dependence over four decades. While conducting controlled strain rate indentations combined with CMX loading, the best approach is to test the material at high strain rates, as small strain rates must be explored through reference creep testing. The small standard deviation of the recorded hardness allows for the identification of minor differences between the constant strain rate and the strain rate jump experiments. Future work in this area is needed to understand these differences, which might help to further characterize the PMMA time-dependent behavior. Moreover, the use of an xSol® heating stage would further extend the parameter space through a uniform microenvironment of a controlled temperature and gas composition around the indenter tip and the sample. Such experiments would mimic in-operando conditions for typical PMMA applications and identify possible limitations of the material ahead of time.
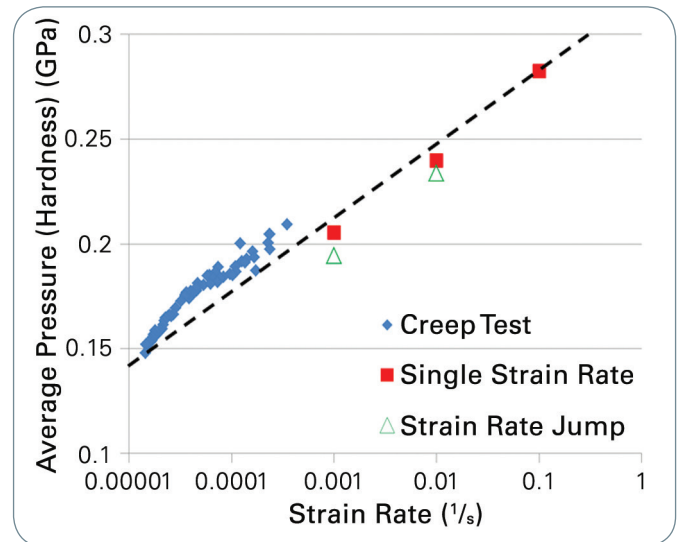

Figure 4. Hardness versus depth for the constant strain rate and strain rate jump tests.

References

1. Lucas, B.N. et al. 1996, *MRS Proceedings*, Volume 436, 1996.

2. S. Asif and J.B. Pethica, *MRS Proceedings*, Volume 505, 1997.

3. S. Asif and J.B Pethica, *Phil. Mag A, Volume* 76, 1105, 1997.

4. Meyer, V. et al., *Mater. Res.*, Volume 26, No. 11, 2011.

Author

Ude D. Hangen (ude.hangen@bruker.com),
Bruker Nano Surfaces Division