Time Dependent Deformation Behavior of PMMA
Creep Testing, Constant Strain Rate, and Strain Rate Jump Indentation

Introduction

Time dependent deformation of materials is typically not investigated by hardness/modulus measurements and the effects on the comparability of the measurement are controlled by using comparable load functions. There are many different material cases where the effects of a changing time scale are negligible and therefore, testing at constant strain rate is not a first concern when performing hardness tests. The first reports of time dependent deformation behavior during instrumented indentation, as well as a concept of analyzing said behavior, was observed on metals with low melting points which exhibit a significant amount of creep (Indium at RT; Lucas et al. 1997). Other metals that exhibit creep at RT include Aluminum and Copper. With current developments in high temperature indentation experiments, where the melting point of various metals is approached, constant strain rate testing becomes increasingly applicable.

Another class of materials that is under investigation is polymers, which undergo creep deformation. Here, the time dependent behavior can be described by their viscoelastic behavior tested in a nanoDMA® III experiment or by a description of their creep behavior (for those polymers which show a low loss). While the creep behavior monitors the deformation velocity of a material with respect to the applied stress, and is typically used to characterize material behavior at very small deformation speeds, 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 the deformation speed range of $1^{1/4}$ to $0.001^{1/4}$, while creep experiments explore the deformation speed of $0.001^{1/4}$ to $0.00000^{1/4}$ and lower (S.A. Asif et al.).

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, $l$. In an indentation experiment, the deformation speed cannot be related to the sample size and therefore the only length scale available for nanoindentation is the penetration depth of the indenter, $h_c$. The strain rate, $dε/dt$, is then defined as $dh/dt/h_c$, although the indentation experiment is not strictly comparable to a tensile test. Since the indentation depth, $h_c$, is always very small at the beginning, it becomes clear that the strain rate can be very high during the initial indentation.

Assuming a sample with constant hardness, it was shown that a constant strain rate experiment could be conducted with a Berkovich indenter (Lucas et al.). Under these preconditions, a strain rate $dh/dt/h_c = \text{const}$ would be fulfilled if the force, $P$, would change in a similar way, $dP/dt/P = \text{const}$. This condition results in an exponential load function for the indentation. Recently, Meyer et al. have shown, on nanocrystalline Al, that the strain rate can be changed during the indentation experiment. The stress level necessary to achieve a speed of deformation is represented by the hardness - the average pressure under the indenter. It was found that the hardness of nanocrystalline Al changes if changing strain rates are used in one load function. A hardness value for strain rate is reached after the exponential loading that is needed to achieve said strain rate is performed for a certain amount of time. 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

A PMMA sample was tested by indentation experiments with different strain rates (0.11/s, 0.011/s, and 0.0011/s) and by strain rate jump experiments. The strain rate jump experiments started at a strain rate of 0.11/s. At a load of 1900 µN, the deformation speed was changed to 0.011/s and 0.0011/s. Additionally, a reference creep test was performed over a time frame of 8000s in order to study the strain rate of PMMA at strain rates between 0.0011/s and 0.000011/s. The creep experiment was performed after a fast loading of the indenter to 9000 µN.

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 strain rate. The curves represented here are the average curves of 4 indentation experiments, which also indicate the reproducibility of the indentation tests.

The modulus shown in Fig. 3 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 is only shown here to confirm that the experiments have been conducted properly.

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 (Fig. 4).

Figure 4: Hardness vs. Depth for the constant strain rate and strain rate jump tests.

Conclusion

Three different tests were performed to study the strain rate behavior of PMMA using a TI 950 TriboIndenter® equipped with nanoDMA® III. Fig. 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 4 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 results allows for the identification of small differences between the constant strain rate and the strain rate jump experiments. Future work in this area is needed to understand these differences, as they might help to further characterize the PMMA time dependent behavior.

Moreover, the use of an xSol 600 heating stage would further extend the parameter space by controlling a small, uniform microenvironment of a controlled temperature and gas composition around the indenter tip and the sample. The aim is to mimic conditions that PMMA faces when used in application and to identify possible limitations of the material, ahead of time.

References: