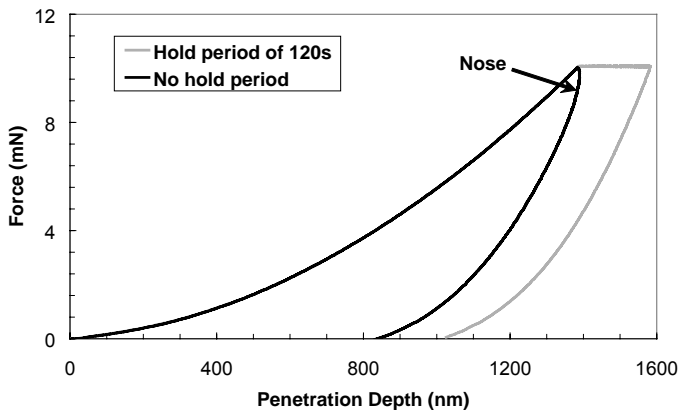


# APPLICATIONS BULLETIN

## *Investigation of creep behaviour using Micro or Nano Indentation Tester (MHT/NHT)*

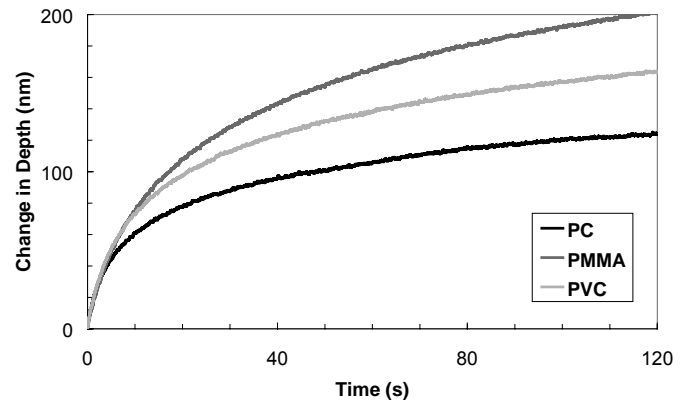
The phenomenon of creep is often observed in indentation testing for certain materials such as polymers and soft metals. Creep depends on the material and normally decreases to very low values within some seconds. Nevertheless, it influences the maximum depth and the unloading curve in a way that non negligible errors of modulus and hardness calculations may occur. This application note focuses on the investigation of creep behaviour for the following amorphous polymers: Polymethylmethacrylate (PMMA), Polycarbonate (PC) and Polyvinylchloride (PVC).

In an indentation test, creep often manifests itself as a bowing out or “nose” in the unloading portion of the force-displacement curve, as shown on the curve without hold period in Fig. 1. For such material, when the force is held during a certain time at the maximum force, the indenter continues to penetrate, as shown in Fig. 1.



**Figure 1:** Force-displacement curves for PMMA. The curve without hold period at maximum force shows a nose in the upper part of the unloading curve whereas the curve with a 120 s hold period shows that the indenter continues to penetrate in the material.

The most common method of measuring creep is to maintain the applied force at a constant maximum value and measure the change in depth of the indenter as a function of time. The relative change of the indentation depth is referred to as the “creep” of the specimen material. Fig. 2 shows a creep comparison for the three amorphous polymers (PMMA, PVC and PC) tested with the same test parameters. PMMA is the sample exhibiting the highest creep followed by PVC and PC.

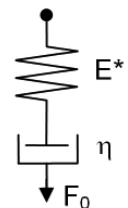


**Figure 2:** Creep comparison of different amorphous polymers from 10 mN indents with 20 mN/min loading rate.

In a previous Application Bulletin (N°18) it has been shown that the indentation creep can be easily determined using the CSM Instruments Micro and Nano Indentation Tester (MHT and NHT). The indentation creep coefficient is defined as the relative change of the indentation depth whilst the applied force remains constant.

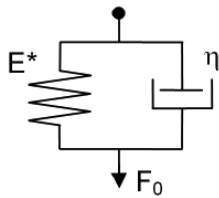
For the investigated polymers, a more empirical approach that yields information about the viscoelastic properties of the specimen is available through mechanical modelling. By modelling the sample to a Maxwell two-element model, the creep response (change in depth over time) at constant force can be expressed according to the following formula:

$$h^2(t) = \frac{\pi}{2} F_0 \cot \alpha \left[ \frac{1}{E^*} + \frac{1}{\eta} t \right]$$



where  $F_0$  is the maximum load,  $\alpha$  is the cone semi angle,  $E^*$  and  $\eta$  are fit parameters representing the bulk and shear moduli of the spring element, and the viscosity term that quantifies the time dependent property of the material, respectively.

Another common model used is the Kelvin two-element model; the creep response then becomes:

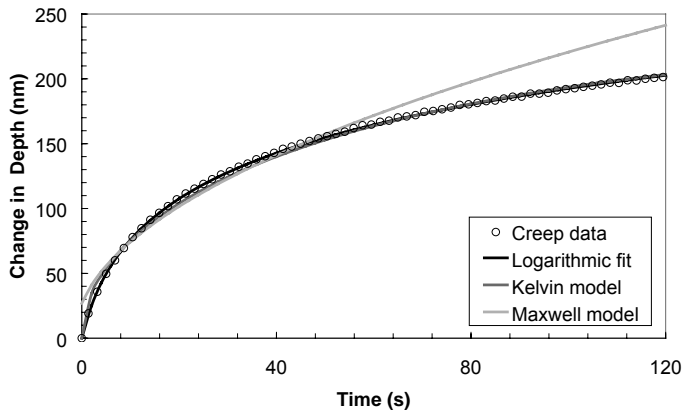
$$h^2(t) = \frac{\pi}{2} F_0 \cot \alpha \left[ \frac{1}{E^*} \left( 1 - e^{-\frac{t E^*}{\eta}} \right) \right]$$


Another model commonly used for soft metals can be used to fit the creep curve with the following logarithmic formula:

$$h(t) = A \ln(Bt + 1)$$

where A and B are fit parameters which depend on temperature, dislocation density, Burgers vector and yield strengthening.

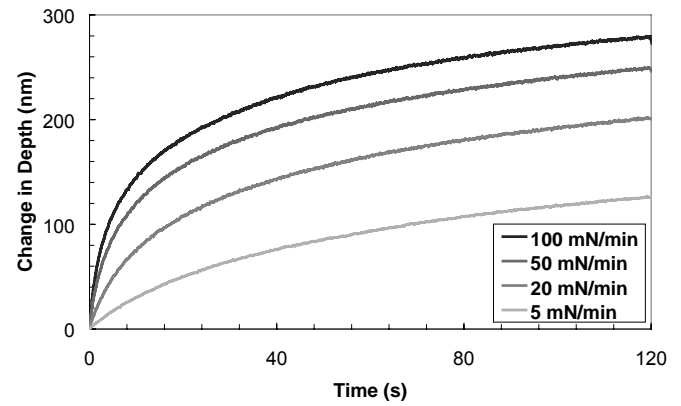
To fit the creep curve to a measurement data file, judicious adjustment of  $E^*$  and  $\eta$ , or A and B need to be determined. The creep curve of PMMA has been investigated with all aforementioned creep models to verify the precision of the model compared to the measurement data, as shown in Fig. 3. The model giving the closest fit to the data is the logarithmic followed by the Kelvin model; the Maxwell model hardly follows the creep data.



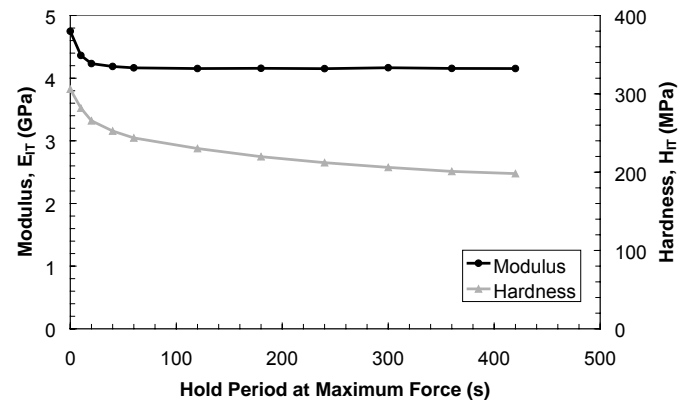
**Figure 3:** Comparison of different fit models on PMMA creep from 10 mN indents with 20 mN/min loading rate.

### Creep influence on hardness and modulus results

The creep behaviour is highly influenced by the loading rate used during the test. Fig. 4. shows the result of creep curves for different loading rates. The faster the loading rate, the higher the creep. Therefore, as the creep is closely connected with plastic deformation, every step within the plastic regime during the indentation test is accompanied by a small amount of creep. Thus, the maximum indentation depth used for the hardness and modulus calculation is strongly affected by the loading rate and the duration of the hold period of the test. Investigation of the modulus and hardness as a function of the hold period has been performed on PMMA, as shown on Fig. 5. These results show that, in that case, only after around 40 s or more can the modulus error due to creep be neglected. Finally, the hardness is even more influenced by creep than the modulus on that sample. The hold period needs to be at least 100 s to avoid the creep effect.



**Figure 4:** Influence of the indentation loading rate on the creep curve for PMMA for 10 mN indents with 120 s hold period.



**Figure 5:** Modulus and hardness as a function of the hold period at the maximum force for PMMA for 10 mN indents with 20 mN/min loading rate.



This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

Editors Nicolas Conté  
Dr Nicholas X. Randall

Should you require further information, please contact:

CSM Instruments  
Rue de la Gare 4  
CH-2034 Peseux  
Switzerland

Tel: + 41 32 557 5600  
Fax: +41 32 557 5610  
info@csm-instruments.com  
www.csm-instruments.com