Design, fabrication and testing of rheological nano –indenter.

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Demands of high throughput in nanoimprint lithography (NIL) dictate to make glass transition temperature of polymer matter used in NIL rather low therefore an approach for measurement of viscous properties of tailored polymers should be developed. The method should allow measuring the properties at the state (films of 100-500nm) in which the polymer is intended to be used. On the other hand it would be highly desirable the method would allow characterizing polymer curing and mechanical properties of the ***cured*** polymer, which planned to use as cost-effective stamps. One more additional usage of the method could be characterization on adhesion properties of polymer surfaces before and after special treatment predicting release properties of stamps as function of temperature.

To meet the requirements a special tool called rheological nano-indenter was designed and fabricated consisting of several components:

a) generic AFM device (see Figure 1), b) original stage (made of ***invar*** to decrease thermal expansion) with heater for *local* heating of a sample at place of indentation, c) original control software to provide complex tip trajectory and flexibility in data acquisition and treatment.

Main measurement action (measuring so called "loading curve", Figure 2) takes only 0.1s. Example of the loading curves is presented in Figure 2. Main characteristics acquired are slopes of loading curve (compliance) as function of temperature. Temperature dependence the compliance for exposed positive resist (950K PMMA) at different doses is shown as an example in Figure 3. Common feature is constant value of the compliance at low temperatures what corresponds to low viscosity (the compliance is equal to inverse stiffness of a cantilever) then the compliance increases what corresponds to viscosity decreasing and to plastic deformation of polymer matter. It is expected that higher exposure results to decrease of molecular weight and it is clearly seen the plastic deformation in accordance to expectations starts at lower temperatures correspondently. One more example illustrates investigation of curing kinetics (Figure 4), thermo-curable polymer was backed at 150C for different times, it is seen that there is no noticeable cross-linking for backing time up to 60min, it is seen also that cross-linking mainly finishes after 120min of backing.

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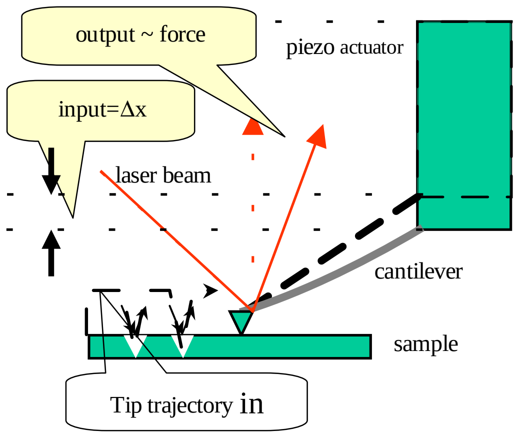


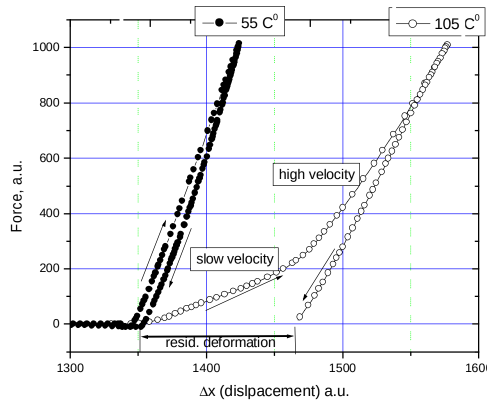
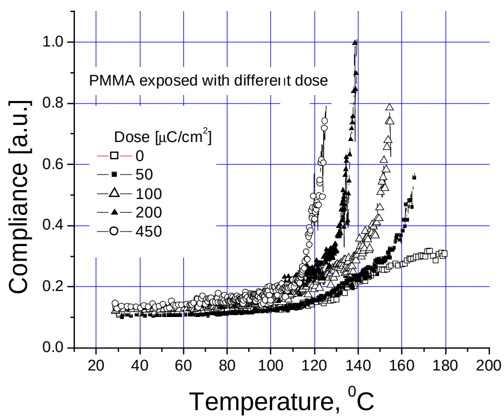


Figure 1 Principal schema of generic AFM and sew-mode of indentation. Elongation (displacement Δx) of actuator is input signal, force measured as cantilever bending is output signal resulting to "loading curve".
Figure 2 Examples of loading curves as function of temperature. At low temperature (55^{0}C) the curve shows elastic behaviour (compliance is very small and does not depend on velocity of the tip), at higher temperature compliance becomes higher demonstrating inelastic behaviour (depending on tip velocity).



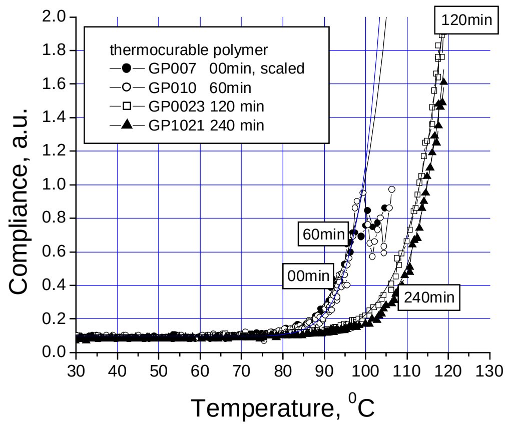


Figure 3 Compliance of 950K PMMA film subjected to different exposures as function of temperature, higher exposures result to more plastic behaviour. In principal distribution of exposure could be measured with nanometer accuracy.
Figure 4 Compliance of a thermocurable resist baked for different times at 150^{0}C. Noticeable hardening (due to cross-linking) occurs after 60min and finishes after 120min of backing. Photocurable polymers can be investigated in similar manner.