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An Advanced Dynamic Microindentation System to Determine Local Viscoelastic Properties of Polymers

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Objectives:

Indentation and dynamic mechanical analysis (DMA) are methods to determine mechanical and viscoelastic material properties. However, indentation only measures local and DMA bulk properties. Combining indentation and DMA broadens the capability of a conventional DMA. The aim of this study is the development of a DMA-Microindentation system to measure the bulk and local viscoelastic properties within one instrument.

Materials and Methods:

Samples made of polycarbonate (PC), polybutylene terephthalate (PBT), annealed / unannealed high density polyethylene (HDPE) and thermoplastic polyurethane (TPU) were investigated by micro-indentation with respect to their local viscoelastic properties using a fine tipped tungsten needle indenter and 3 diamond indenters with Berkovich, Vickers and Rockwell geometry.

The indenters were implemented in a DMA 242 C (Netzsch, Germany) with special indenter holders, Fig. 1.

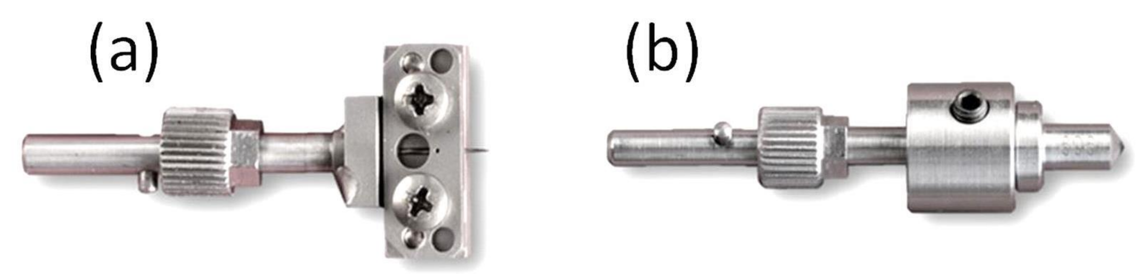

Fig 1: Special indenter holders for DMA, (a) cone indenter (b) diamond indenters [1]

The measurements were performed using dynamic microindentation in the DMA penetration mode to determine the complex modulus E* at room temperature. After ensuring contact of the indenter with the sample surface and setting a zero position, a force-controlled segment was performed with an amplitude of 120 µm and load of 1.25 N, with frequency 1 Hz. The results were compared to the corresponding complex moduli of all polymers determined by 3-point bending at room temperature, the frequency 1 Hz, maximum load of 5 N and a maximum amplitude of 25 µm. Furthermore, values from literature were taken to complete the comparison. A special x-y-stage with a laser positioning system was developed and adapted to the DMA to perform spatially resolved measurements, Fig. 2.

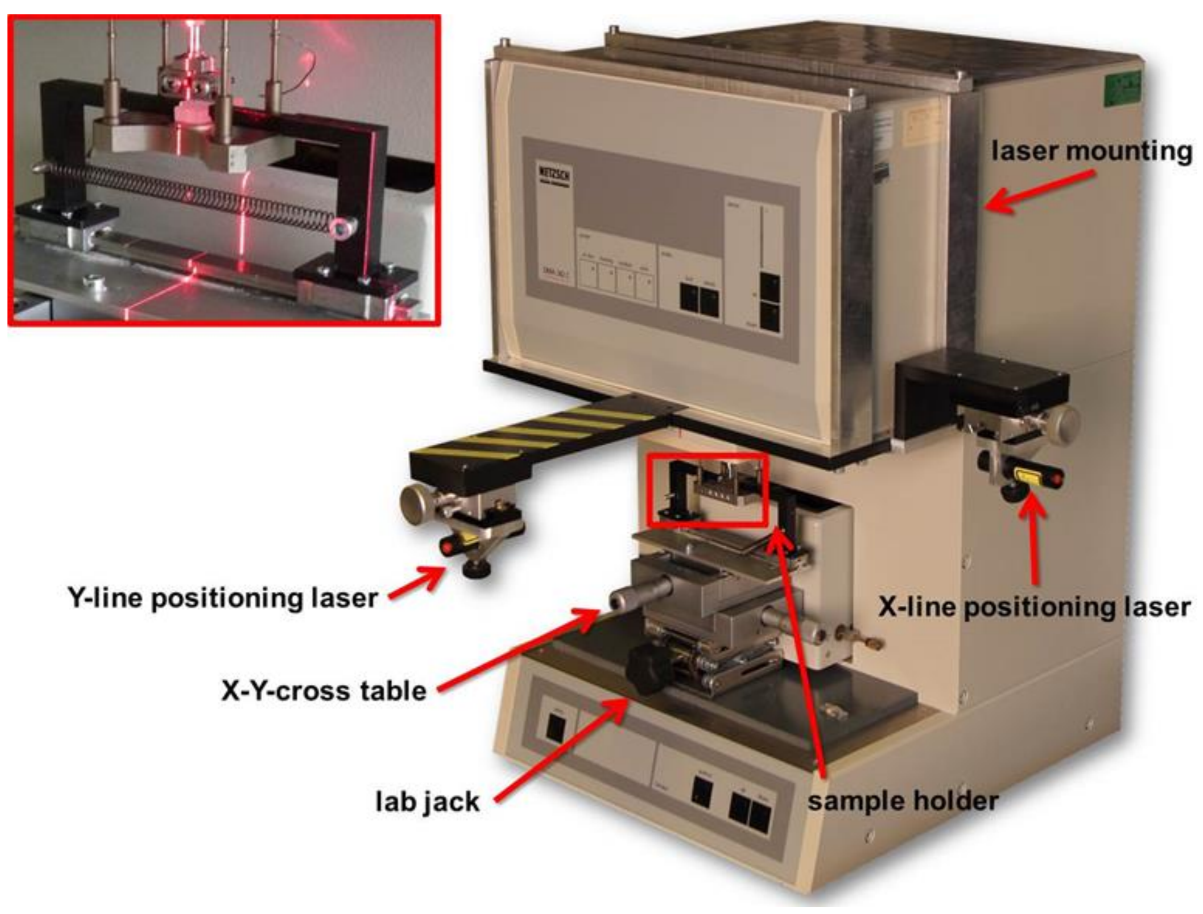

Fig 2: Specially developed x-y-stage for spatial resolution of microindentations[2]

Results and Discussion:

With known indenter geometry factors [1], the complex modulus can be calculated with [3,4]:

$$E^* = \frac{F_s^*}{A_s^*} \times \frac{(1-\upsilon_s^2)}{2} \times \frac{\sqrt{\pi}}{\sqrt{A}} \qquad (1)$$

F_s^* measured complex load [N]
A_s^* measured complex amplitude [µm]
v sample Poisson's ratio
A contact area of indenter [µm]

Although for PBT the diamond indenters underestimated the 3-point bending and literature values, a comparison of the measured complex moduli of PC, HDPE, and TPU show a quite good agreement with the bulk values, Fig. 3. Since the 3-point bending moduli and literature values, which originate from tensile testing, represent bulk mechanical properties, some deviation between the literature values and the local viscoelastic complex moduli can be expected.

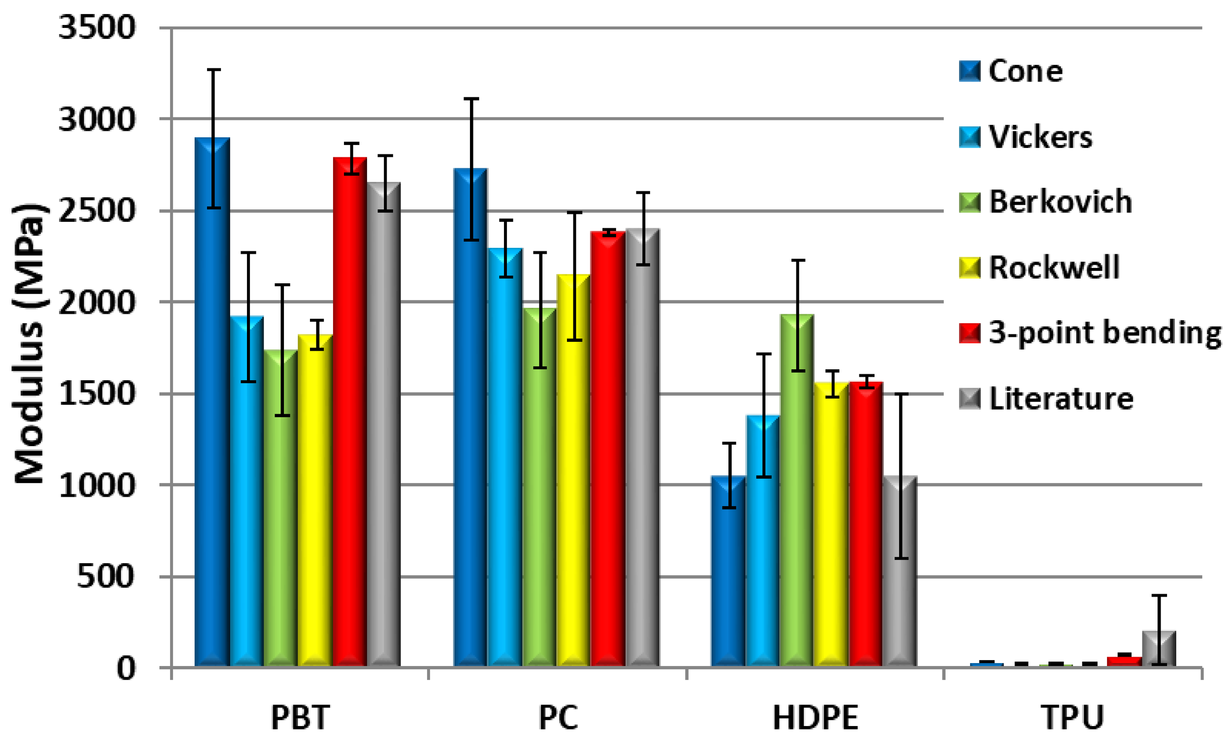

Fig 3: Complex moduli for investigated polymers obtained with various indenters, three-point bending, and Young's moduli from literature[5]

The reproducibility of the cone indenter was evaluated on a neat injection molded PC plate. The mean values of the complex moduli of 20 dynamic microindentations of unannealed and annealed PC were (3217 ± 729) MPa and (2402 ± 253) MPa, respectively. The result of the unannealed sample obviously shows that stresses caused by processing has an immense influence on the outcome compared to the annealed sample, where these processing stresses were (partially) relaxed during annealing. The standard deviation of both mean values also indicates that the effect of these processing stresses can locally differ enormously.

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Another highlight of the development of this microindentation system is the possibility to spatially resolve dynamic microindentations. This is displayed on the cross-section surface of the narrow parallel area in the middle of a unannealed injection molded HDPE tensile bar with the fine tipped cone indenter, Fig. 4.

Close to the edges of the sample, near 0 and 4000 µm, the complex moduli show lower values than in the middle of the cross-section. Due to the fast cooling rates close to the mold, fewer crystalline regions were formed near the edges and, therefore, the lower complex moduli were measured. However, in the center of the cross-section, the cooling rates were slower, and thus, more crystalline regions were formed, leading to the higher complex moduli.

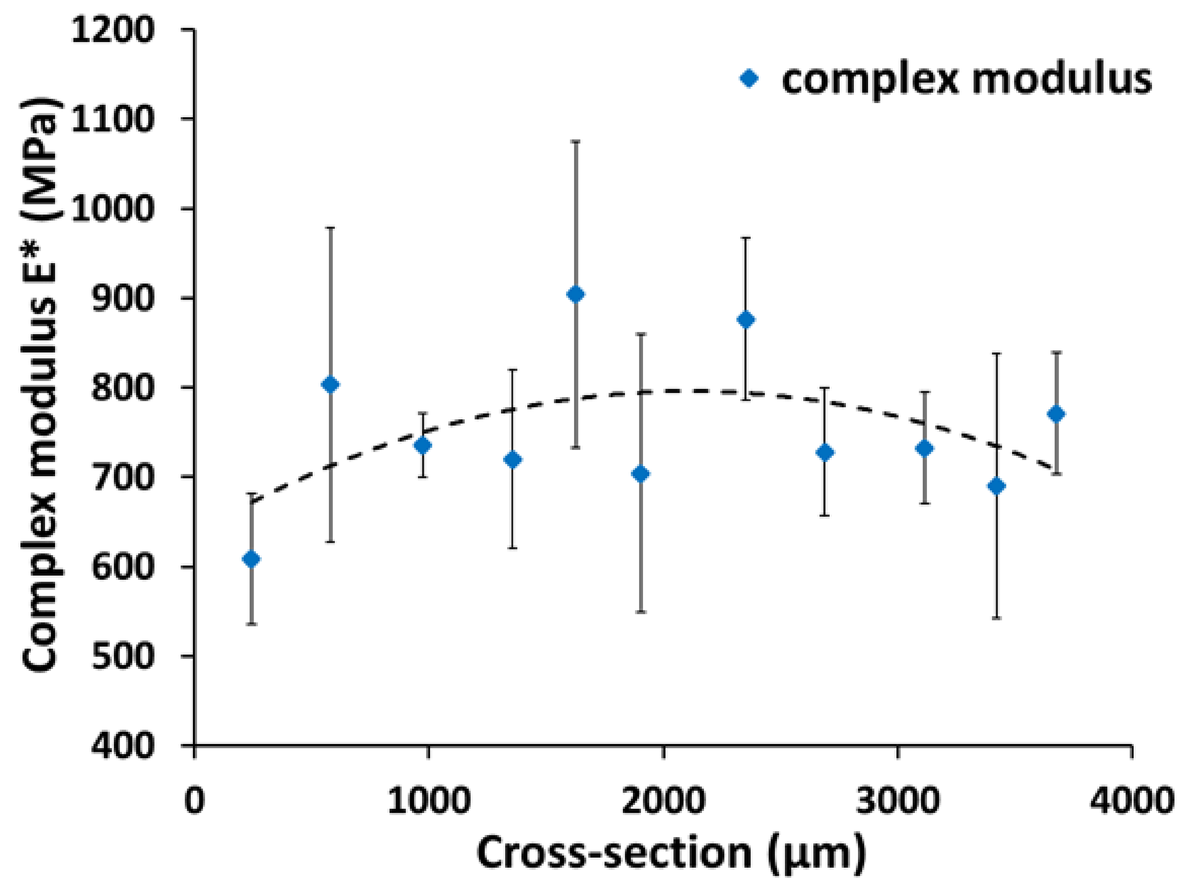

Fig 4: Complex modulus across the cross-section of unannealed HDPE; second order polynomial fit as a trend (dotted) line.[2]

Conclusions:

It is shown that different polymers and small inhomogeneous mechanical properties distribution can be intercepted and distinguished by the proposed microindentation system. A comparison of the complex moduli measured by dynamic microindentation with fine tipped tungsten cone indenter, different diamond indenters, 3-point bending, and literature overall reveals a good agreement. Additionally, dynamic microindentations with the cone indenter on the cross-section surface of the unannealed HDPE tensile bar with the specially developed x-y-stage showed that small local differences in mechanical properties can be spatially resolved.

This newly developed DMA-microindentation system allows for the determination of bulk AND local viscoelastic properties within one conventional DMA-instrument.

References:

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[5] Datasheets of tested polymers

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